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(54) Title
 FABRIC SCENTING METHOD

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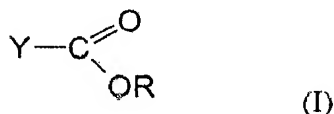
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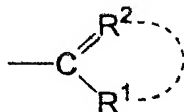
(57) Claim

1. A process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, said process being characterized in that said detergent and optionally said fabric softener contains a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener is obligatorily used and contains a compound of formula

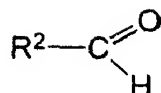


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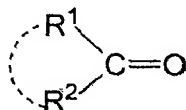
- a. R represents a radical derived from a fragrant alcohol of formula ROH and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6; or
- b. Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula



wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from a fragrant aldehyde of formula

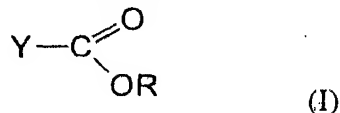


or R^2 represents an alkylidene radical and R^1 an alkyl radical, R^1 and R^2 being then derived from a fragrant ketone of formula



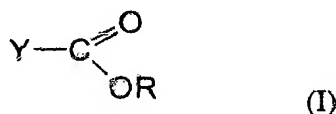
and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.

5. A process for prolonging the diffusion effect of the odour developed by fabrics and which is characteristic of a fragrant alcohol of formula ROH , said process being characterized in that the fabrics undergo a washing cycle in the presence of a lipase-containing detergent and, optionally, a subsequent treatment with a fabric softener, said detergent and optionally said fabric softener containing a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener being obligatorily used and containing a compound of formula



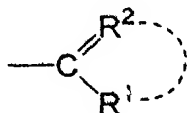
wherein R represents a monovalent radical derived from said fragrant alcohol, and Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical, or a $-(\text{CH}_2)_n\text{COOR}$ group wherein R is defined as above and n is an integer from 0 to 6.

13. A compound of formula



wherein

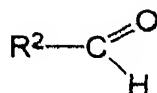
- R represents a radical derived from an fragrant alcohol of formula ROH and Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical, or a $-(\text{CH}_2)_n\text{COOR}$ group wherein R is defined as above and n is an integer from 0 to 6; or
- Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula



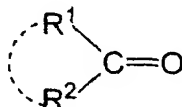
wherein, either R^1 represents hydrogen and R^2 represents an alkylidene radical derived from a fragrant aldehyde of formula

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or R^2 represents an alkylidene radical and R^1 an alkyl radical, R^1 and R^2 being then derived from a fragrant ketone of formula



and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.



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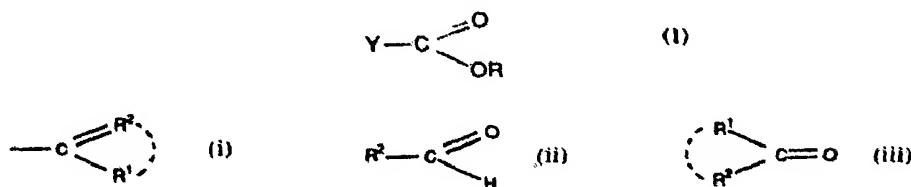
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(54) Titre: FABRIC SCENTING METHOD

(54) Titre: PROCEDE DE PARFUMAGE DE TEXTILES



(57) Abstract

A method for scenting fabrics being washed in the presence of a lipase-containing detergent, whereafter they are optionally treated with a fabric softener. The method is characterised in that said detergent and/or said fabric softener contain a compound of formula (I), wherein (a) R is a radical derived from an odoriferous alcohol of formula ROH, and Y is a saturated or unsaturated, straight or branched C₇₋₂₄ alkyl radical, or a group -(CH₂)_nCOOR wherein R is as defined above and n is an integer of 0-6; or (b) Y is a saturated or unsaturated, straight or branched C₇₋₂₄ alkyl radical, and R is a group of formula (i), wherein either R¹ is hydrogen and R² is an alkylidene radical derived from an odoriferous aldehyde of formula (ii), or R² is an alkylidene radical and R¹ is an alkyl radical, R¹ and R² being derived from an odoriferous ketone of formula (iii), and optionally forming part of an optionally substituted ring containing 5-18 carbon atoms, as shown by the dotted line. The method is advantageous in that it provides slow release of said odoriferous alcohol, aldehyde or ketone and thereby prolongs the fabric scenting effect thereof.

Applicant :

FIRMENICH SA, Geneva

Applicant's reference:

4260-PCT

Title of the invention :

Fabric Scenting Method

**MIG/AIB/BLD
09.02.95**

Fabric Scenting Method

Technical field and prior art

5 The present invention relates to the field of perfumery and, more particularly, to improved perfumed detergents and fabric softeners.

 The use of enzymes in fabric detergents, in order to improve their efficiency, has been known for a number of years. Amongst said enzymes, lipases are particularly preferred, as a result of their capacity to hydrolyse
10 the fat materials on the dirty linen and thus to facilitate its cleaning. However, it is known that malodour problems can occasionally occur under certain application conditions. In order to solve these malodour problems, there has been proposed a method (see for example EP 430 315) consisting in carefully choosing the perfuming ingredients incorporated in
15 the detergents and which, following the washing, are deposited on the fabrics. Therefore, appropriate perfuming of said detergents appears to be extremely important.

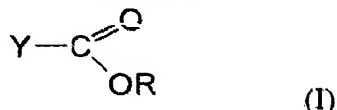
 On the other hand, it would be desirable that said detergents and fabric softeners are able to impart to the fabrics a long-lasting odour, such
20 that the user perceives this odour even a long time after the textiles have been washed and subsequently dried. To this end, it has been known to use in the fabric detergents and softeners perfuming ingredients which have a good tenacity on the fabrics, i.e. ingredients whose odour, once imparted to the textiles upon the washing, can then be perceived by the consumer for
25 several days. However, there are many perfuming substances known for their extremely pleasant odours, and namely a quality of "freshness" often associated with the notion of cleanliness, which substances are unfortunately not very tenacious, or even not tenacious at all, on fabrics, such that their perfuming effect can only be perceived very briefly, at the
30 most for a few hours following the washing and drying operations. Clearly, prolonging the fragrance effect of such substances, and thus the "freshness" of the fabrics, would be highly desirable.

 The present invention brings precisely a novel solution to this problem. We have now unexpectedly discovered a better process for
35 perfuming textiles washed with detergents containing lipases. We have in fact been able to establish that, by adding particular ingredients to the fabric detergent and/or to the fabric softener that is subsequently applied, one could distinctly improve the odour of the fabrics treated with these

products and prolong in a remarkable way the fragrance of said fabrics after drying.

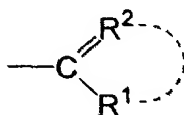
Description of the Invention

A first object of the present invention is thus to provide a process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, said process being characterized in that said detergent and optionally said fabric softener contains a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener is obligatorily used and contains a compound of formula

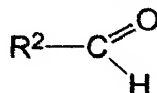


wherein

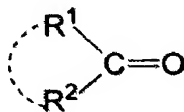
- a. R represents a radical derived from a fragrant alcohol of formula ROH and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6; or
- b. Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula



wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from a fragrant aldehyde of formula



or R² represents an alkylidene radical and R¹ an alkyl radical, R¹ and R² being then derived from a fragrant ketone of formula



and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.



By an alkylidene radical derived from a fragrant aldehyde or ketone, it is understood here a radical which, upon the conversion of enol-ester (I) into said aldehyde or ketone, regenerates the corresponding R group which is the substituent of said aldehyde or ketone. Thus, for example, when the
5 fragrant aldehyde is 3,7-dimethyl-6-octenal (R is 3,7-dimethyl-6-octenyl), the corresponding alkylidene radical in enol-ester (I) is 3,7-dimethyl-1,6-octadienyl.

According to a variant of the invention, there is provided a process for perfuming fabrics washed in the presence of a lipase-containing
10 detergent, which process comprises the treatment of said fabrics, after the washing cycle, with a fabric softener containing a compound of formula (I) as defined above.

By "fragrant alcohol", "fragrant aldehyde" or "fragrant ketone" it is meant here any alcohol, aldehyde, respectively ketone, of current use in
15 perfumery, which is capable of imparting an odour to fabrics or textiles, upon the process of washing and/or the treatment with a fabric softener. We have in fact discovered that the perfuming effect of such compounds could be remarkably improved, and namely their diffusion prolonged, if they were replaced by the corresponding compound (I) in the detergent or
20 fabric softener used.

This result is all the more surprising in that the compounds (I) are themselves either devoid of odour, or they possess weak and characterless odours, and are thus apparently useless for perfumery. Yet, when used according to the invention, they are not only capable of
25 imparting to the fabrics the characteristic odour of the corresponding alcohol, aldehyde or ketone, but also of prolonging their effect of diffusion, such that said odour develops itself for much longer periods of time than in the case where said corresponding alcohol, aldehyde or ketone is directly added to the detergents or fabric softeners. Therefore, the use of compounds
30 (I) according to the process of the invention is translated into an enhanced substantivity of the corresponding alcohols, aldehydes and ketones.

The advantages of this process are all the more obvious in the case of the numerous odoriferous alcohols which are known to be weakly tenacious on washed fabrics and the odour of which, although distinctly
35 perceived when the fabrics are removed from the washing machine, does not remain on the linen and can no longer be perceived after 12 to 24 h.

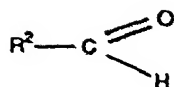
Many examples of such alcohols can be found in the prior art, some of which only contribute to the odour of the fabrics for a very short

time, and no doubt many others will be discovered in the future. For all these alcohols, the process according to the invention makes it possible to remarkably improve their odoriferous performance on fabrics, by prolonging the diffusion time of their characteristic notes and thus the time during which they contribute significantly to the overall odour. We have in fact established that the use of the corresponding compounds (I) in the detergents and/or fabric softeners made it possible to obtain a perfuming effect equivalent to the one that would have been observed with the prolonged diffusion ("slow-release") of the alcohol, if such a diffusion would have been possible, which is not the case in practice.

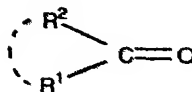
Quite clearly, it is impossible to list in an exhaustive manner all the alcohols of formula ROH known to this day, which are capable of imparting pleasant odours to the textiles treated with the laundry products perfumed by way of said alcohols, and the perfuming effect of which can be remarkably improved according to the invention. However, by way of example, one can cite alcohols such as anisic alcohol, cinnamic alcohol, fenchylic alcohol, 9-decen-1-ol, phenethylol, citronellol, 3-methyl-5-phenyl-1-pentanol (origin: Firmenich SA, Geneva, Switzerland), Mayol® (7-p-menthan-1-ol; origin: Firmenich SA, Geneva, Switzerland), dihydromyrcenol (2,6-dimethyl-7-octen-2-ol), geraniol (3,7-dimethyl-2,6-octadien-1-ol), (Z)-3-hexen-1-ol, 1-hexanol, 2-hexanol, 5-ethyl-2-nonanol, 2,6-nonadien-1-ol, borneol, 1-octen-3-ol, 4-cyclohexyl-2-methyl-2-butanol (origin: Firmenich SA, Geneva, Switzerland), 2-methyl-4-phenyl-2-butanol, 2-methyl-1-phenyl-2-propanol, cyclomethylcitronellol, decanol, dihydroeugenol, 8-p-menthanol, 3,7-dimethyl-1-octanol, 2,6-dimethyl-2-heptanol, dodecanol, eucalyptol, eugenol, Florol® (tetrahydro-2-isobutyl-4-methyl-4(2H)-pyranol; origin: Firmenich SA, Geneva, Switzerland), isoeugenol, linalol, Tarragol® (2-methoxy-4-propyl-1-cyclohexanol; origin: Firmenich SA, Geneva, Switzerland), terpineol, tetrahydromuguol, 3,7-dimethyl-3-octanol and Lyrall® (3 and 4-(4-hydroxy-4-methylpentyl)-cyclohex-3-ene-1-carbaldehyde; origin: International Flavors and Fragrances, USA).

It is quite obvious, however, that the process of the invention is perfectly general and can relate to many other alcohols which the skilled person is quite able to choose from the general knowledge in the art and as a function of the olfactive effect it desires to achieve.

Analogous considerations apply to the aldehydes of formula



and to the ketones of formula



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The process of the invention is advantageous whenever the compounds such as those mentioned above show a weak tenacity on fabrics, and there are many odoriferous aldehydes and ketones whose performance on
10 textiles turns out to be distinctly improved when these compounds exert their perfuming activity by way of the corresponding enol-esters of formula (I).

Again, although one cannot cite in an exhaustive manner all the
15 fragrant aldehydes and ketones which can be used according to the process of the invention, there can be cited, by way of example, compounds such as C₆ to C₁₂ aldehydes, hydratropic aldehyde, methyl nonyl acetaldehyde, phenylpropanoic aldehyde, Acropal® [3- or 4-(4-methyl-3-pentenyl)-3-cyclohexene-1-carbaldehyde; origin: Givaudan-Roure, Vernier, Switzerland], 2-methyldecanal, 4-isopropyl-1-benzeneacetaldehyde, (4-
20 methyl-1-phenyl)acetaldehyde, Z-6-nonenal, citral, citronellal, 9-decenal, 3-(4-isopropyl-1-phenyl)-2-methylpropanal (origin: Firmenich SA, Geneva, Switzerland), (E,E)-2,4-heptadienal, (E,E)-2,4-nonadienal, (E,E)-2,4-decadienal, 5,9-dimethyl-4,9-decadienal, (Z)-6-octenal, Farenal® (2,6,10-trimethyl-9-undecenal; origin: Givaudan-Roure, Vernier, Switzerland),
25 Foliaver® [3-(4-methoxy-1-phenyl)-2-methylpropanal; origin: International Flavors and Fragrances, USA], Heliopropanal® [3-(1,3-benzodioxol-5-yl)-2-methylpropanal; origin: International Flavors and Fragrances, USA], (Z)-4-heptenal, le 3,5,5-trimethylhexanal (origin: International Flavors and Fragrances, USA), (4-methyl-1-
30 phenoxy)acetaldehyde, hydroxycitronellal, isocyclocitral (origin: International Flavors and Fragrances, USA), Lilial® [3-(4-tert-butyl-1-phenyl)-2-methylpropanal; origin: Givaudan-Roure, Vernier, Switzerland], le 1-p-menthene-9-carbaldehyde (origin: Firmenich SA, Geneva, Switzerland), Lylal® [3- and 4-(4-hydroxy-4-methylpentyl)-3-
35 cyclohexene-1-carbaldehyde; origin: International Flavors and Fragrances, USA], 2,6-dimethyl-5-heptenal, 1-p-menthen-9-al, (E)-2-octenal, (2E,6Z)-2,6-

nonadienal, 3-methyl-5-phenylpentanal, (E)-4-decenal, (E)-2-undecenal, 3,7-dimethyloctanal, Zestover (2,4-dimethyl-3-cyclohexene-1-carbaldehyde; origin: Firmenich SA, Geneva, Switzerland), 3-phenylbutanal, Scentenal® (octahydro-5-methoxy-4,7-methano-1H-indene-2-carboxaldehyde; origin: 5 Firmenich SA, Geneva, Switzerland), 2,5,9-trimethyl-4,9-decadienal, Intreleven aldehyde (undecenal; origin: International Flavors and Fragrances, USA), 4-methyl-phenyl-propanoic aldehyde, 4-(4-hydroxy-1-phenyl)-2-butanone, benzylacetone, the ionones, 3-(4-tert-butyl-1-phenyl)propanal, carvone, 3,7-dimethyl-1,1-bis(11-methyldodecyloxy)-2,6- 10 octadiene, muscone, 2-pentyl-1-cyclopentanone, l'ethyl amyl ketone, l'ethyl pentyl ketone, la 2-heptyl-1-cyclopentanone, geranylacetone, Iralia® (methylionone; origin: Firmenich SA, Geneva, Switzerland), Iso E super [1-(octahydro-2,3,8,8-tetramethyl-2-naphthalenyl)-1-ethanone; origin: International Flavors and Fragrances, USA], 6-methyl-5-hepten-2-one, 15 methyl jasmonate, methyl hexyl ketone, methyl pentyl ketone, methylnonyl ketone, cis-jasmone, Hedione® (methyl dihydrojasmonate; origin: Firmenich SA, Geneva, Switzerland), civettone, 4-(1,1-dimethylpropyl)-1-cyclohexanone, Exaltone® (cyclopentadecanone; origin: Firmenich SA, Geneva, Switzerland), 2,6,6-trimethyl-2- 20 cyclohexene-1,4-dione, p-tert-butylcyclohexanone, tricyclo[6.2.1.0^{2,7}]undec-9-en-3-one (origin: Firmenich SA, Geneva, Switzerland), 10,10-dimethyl-tricyclo[7.1.1.0^{2,7}]undec-2-en-4-one (origin: Firmenich SA, Geneva, Switzerland), Vertofix coeur (origine: International Flavors and Fragrances, USA), la perhydro-5,5,8a-trimethyl-2-naphthalenone (origin: 25 Firmenich SA, Geneva, Switzerland) or 5-methyl-exo-tricyclo[6.2.1.0^{2,7}]undecan-4-one.

Furthermore, it should be noted that the process of the invention is of much more general use than merely the improved perfuming of textiles treated with laundry products containing lipases. It constitutes, in 30 fact, a general process for washing said textiles which is also advantageous whenever it is desired to improve the activity of certain agents, fragrant or other, currently present in laundry products, i.e. detergents and fabric softeners. It is well-known for instance that said laundry products are often very aggressive media, wherein a great number of perfuming ingredients, 35 and more particularly fragrant aldehydes, turn out to be unstable and cannot therefore be used to perfume those products, nor the textiles washed therewith. The use according to the invention of the corresponding esters and enol-esters of formula (I) can obviate this problem whenever said enol-

esters turn out to be more stable. It is also quite clear that one can make use of the process of the invention to improve and prolong the action of bactericide agents, namely alcohols, whether they are fragrant or not.

Therefore, the process according to the invention is in fact a
 5 general process for treating textiles or fabrics upon the washing of the latter with a lipase-containing detergent, which washing can be followed by treatment with a fabric softener, according to which process any alcohol, aldehyde or ketone currently used in fabric detergents or softeners for its perfuming, bactericide or other activity, can be replaced by the
 10 corresponding compound (I), so as to improve its activity.

Compounds (I) are novel chemical entities and thus are also the object of the invention. Amongst said compounds, those wherein Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical are fatty acids esters or enol-esters which do not themselves possess
 15 an interesting odour, but which, when used according to the process of the invention, are capable of imparting to the fabrics the fragrances typical of the corresponding alcohols, aldehydes and ketones defined above. Said compounds, in particular the derivatives having 12 to 16 carbon atoms, are preferred according to the invention, owing to the particularly
 20 advantageous results obtained upon their use.

As regards the compounds (I) wherein Y represents a $-(CH_2)_nCOOR$ group, R being defined as above and n being an integer from 0 to 6, they are novel diesters, which are either devoid of odour, or possess odours which are not particularly useful for perfumery. However, they
 25 showed similar behaviour to that of the compounds (I) cited here-above, when used in the context of the present invention.

The compounds (I) of the invention can be added to the detergents and fabric softeners either on their own, or in admixture with other perfuming ingredients, solvents or adjuvants, of current use in
 30 perfumery. The concentrations in which they can be added to the detergents and fabric softeners according to the invention have the values usual in the art for this type of products. The skilled person is quite able to select such values as a function of the nature of the product to be perfumed and of the desired olfactive effect. By way of example, concentrations of the
 35 order of 0.01 to 1%, or even up to 5%, by weight of compound (I), relative to the weight of detergent or fabric softener composition, can be cited.

The fabric detergents and softeners according to the invention can take the form of powders or granular solids, bars, pastes or yet aqueous or

non-aqueous liquids, and contain the usual ingredients for this type of product. Thus, the detergents can typically contain, in addition to the lipase (see, for example, EP 430 315 for a detailed description of the type of lipases that can be used according to the present invention), for example active
 5 anionic, cationic, zwitterionic or non-ionic compounds, as well as filling agents, bleaching agents, confining agents and other ingredients of current use in the detergent bases intended for washing linen. A detailed description of the base detergent compositions which are adapted to be used according to the process of the invention is unwarranted here. A great
 10 many examples of such compositions can be found in the art and namely in the literature which is cited for instance in the European patent application mentioned above, or yet in European patent application EP 397 245. By way of example, a base detergent of this type, to which there is added the lipase in the desired concentration, can have the following
 15 composition (origin : Henkel KGaA, Düsseldorf, Germany) :

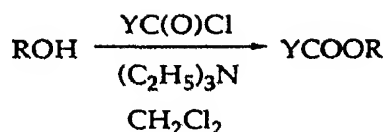
	<u>Ingredients</u>	<u>% by weight</u>
	Linear sodium alkyl benzene	
	sulfonate (average length of the alkane chain : C _{11.5})	8.0
20	Ethoxylated tallow alcohol (14 EO)	2.9
	Sodium soap	
	(chain length C ₁₂₋₁₆ : 13-26%	
	C ₁₈₋₂₂ : 74-87%)	3.5
	Sodium triphosphate	43.8
25	Sodium silicate (SiO ₂ : Na ₂ O=3.3:1)	7.5
	Magnesium silicate	1.9
	Carboxymethylcellulose	1.2
	Sodium ethylenediaminetetraacetate	0.2
	Sodium sulfate	21.2
30	Water	9.8
	Total	100.0

Similar considerations apply to the fabric softener bases according
 35 to the invention, which will typically contain cationic softening ingredients of the type cited in EP 397 245 or in the literature mentioned in this reference.

The compounds of the invention can be prepared by conventional synthetic methods. For example, the derivatives of fragrant

alcohols were prepared by the esterification method summarized in the following reaction scheme :

Scheme I

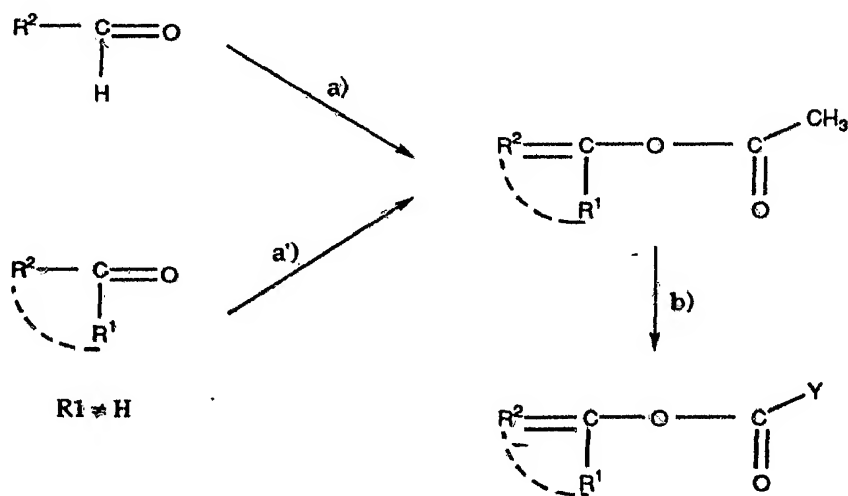


5

wherein Y and R are defined as in claim 1.

The enol-esters of fragrant aldehydes and ketones were also prepared by conventional methods, starting from said aldehydes and ketones, by way of reactions of the type of those represented hereinafter :

10



R¹, R² and Y are defined as in claim 1b.

- 15 a) acetic anhydride ; potassium acetate ; ethylamine ; 120° ; see, for example, D.P. Simmons et al., *Helv. Chim. Acta* **71**, 1000 (1988)
- a') acetic anhydride ; p-toluenesulfonic acid (cat.) ; 120° ; see, for example, T. Taapken et al., *J. Chem. Soc. Perkin Trans. I*, **1994**, 1439
- 20 b) potassium tert-butoxyde ; YCOCl ; see, for example, P. Duhamel et al., *J. Chem. Soc. Perkin Trans. I*, **1993**, 2509

The reaction conditions are described in detail in the preparation examples presented hereinafter, wherein the temperatures are in degrees centigrade and the abbreviations have the usual meaning in the art. The invention will also be illustrated by way of application examples.

25

Embodiments of the invention**Example 1****Preparation of mono-esters****General method**

To a stirred solution of the appropriate ROH alcohol (0.064 mole) and triethylamine (7.4 g, 0.073 mole) in CH_2Cl_2 (110 ml), there was added dropwise during 20 min, at 15° and under N_2 the corresponding $\text{YC}(\text{O})\text{Cl}$ acyl chloride (0.07 mole), wherein Y is a C_7 to C_{24} , linear or branched, saturated or unsaturated alkyl radical. After 2 h at room temperature, the mixture was poured on aq. sat. NaHCO_3 (excess) and the organic phase was separated. Extraction of the aqueous phase with CH_2Cl_2 was followed by washing of the combined organic phases with 10% aqueous NaCl. The reaction product was dried over Na_2SO_4 , concentrated and distilled to provide the desired ester in a pure state and in 80 to 90% yield.

The following mono-esters were prepared according to the above-described general method.

a. 2-phenylethyl octanoate

B. p. $100-101^\circ/4 \text{ Pa}$

$\text{IR}(\text{CHCl}_3)$: 2930, 2858, 1728, 1498, 1455, 1168, 1106 cm^{-1}

$\text{NMR}^1(\text{H}, 360\text{MHz})$: 0.88(t, $J=7\text{Hz}$, 3H); 1.27(8H); 1.59(2H); 2.28(t, $J=7\text{Hz}$, 2H); 2.93(t, $J=7\text{Hz}$, 2H); 4.29(t, $J=7\text{Hz}$, 2H) 7.20-7.35(5H) δ ppm.

$\text{NMR}^{13}(\text{C})$: 173.7(s); 138.0(s); 128.9(d); 128.5(d); 126.5(d); 64.7(t); 35.2(t); 34.4(t); 31.7(t); 29.1(t); 28.9(t); 25.0(t); 22.6(t); 14.0(q) δ ppm.

MS: 248(0, M^+), 127(2), 104(100), 57(12).

b. 2-phenylethyl hexadecanoate

M. p. 40°

$\text{IR}(\text{CHCl}_3)$: 2927, 2855, 1728, 1456, 1174 cm^{-1}

$\text{NMR}^1(\text{H}, 360\text{MHz})$: 0.88(t, $J=7\text{Hz}$, 3H); 1.26(24H); 1.58(2H); 2.27(t, $J=7\text{Hz}$, 2H); 2.93(t, $J=7\text{Hz}$, 2H); 4.29(t, $J=7\text{Hz}$, 2H); 7.20-7.35(5H) δ ppm.

$\text{NMR}^{13}(\text{C})$: 173.7(s); 138.0(s); 128.9(d); 128.5(d); 126.5(d); 64.7(t); 35.3(t); 34.4(t); 32.0(t); 29.7(2t); 29.5(t); 29.4(t); 29.3(t); 29.2(t); 25.0(t); 22.7(t); 14.1(q) δ ppm.

MS : 360(0, M⁺), 104(100).

c. 3,7-dimethyl-6-octenyl octanoate

B. p. 108-109°/2.7 Pa

5 IR(CHCl₃) : 2829, 2858, 1725, 1460, 1379, 1231, 1171, 1106 cm⁻¹

NMR(¹H, 360MHz) : 0.88(t, J=7Hz, 3H) ; 0.91(d, J=7Hz, 3H) ; 1.10-1.80(15H) ; 1.61(s, 3H) ; 1.68(s, 3H) ; 1.98(m, 2H) ; 2.29(t, J=7Hz, 2H) ; 4.10(m, 2H) ; 5.09(broad t, J=7Hz, 1H) δ ppm.

10 NMR(¹³C) : 173.9(s) ; 131.3(s) ; 124.7(d) ; 62.8(t) ; 37.1(t) ; 35.6(t) ; 34.5(t) ; 31.7(t) ; 29.6(d) ; 29.2(t) ; 29.0(t) ; 25.7(q) ; 25.5(t) ; 25.1(t) ; 22.6(t) ; 19.5(q) ; 17.7(q) ; 14.1(q) δ ppm.

MS : 282(0, M⁺), 138(30), 123(56), 109(25), 95(77), 81(100), 69(59).

d. 3,7-dimethyl-6-octenyl hexadecanoate

15 B. p. (bulb-to-bulb distillation) 210-230°/5.3 Pa

IR(CHCl₃) : 2827, 2855, 1725, 1465, 1233, 1178 cm⁻¹

NMR(¹H, 360MHz) : 0.88(t, J=7Hz, 3H) ; 0.92(d, J=7Hz, 3H) ; 1.15-1.35(26H) ; 1.44(m, 1H) ; 1.60(s, 3H) ; 1.68(s, 3H) ; 1.50-1.70(4H) ; 1.98(m, 2H) ; 2.28(t, J=7Hz, 2H) ; 4.10(m, 2H) ; 5.09(broad t, J=7Hz, 1H) δ ppm.

20 NMR(¹³C) : 173.9(s) ; 131.3(s) ; 124.6(d) ; 62.8(t) ; 37.1(t) ; 35.6(t) ; 34.5(t) ; 32.0(t) ; 29.7(2t) ; 29.6(d) ; 29.5(t) ; 29.4(t) ; 29.3(t) ; 29.2(t) ; 25.7(q) ; 25.5(t) ; 25.1(t) ; 22.7(t) ; 19.5(q) ; 17.7(q) ; 14.1(q) δ ppm.

MS : 394(0, M⁺), 138(32), 123(30), 95(60), 82(70), 69(70), 57(100).

25 e. 3-methyl-5-phenylpentyl octanoate

B. p. 133-135°/2.7 Pa

IR(CHCl₃) : 2930, 2858, 1728, 1496, 1456, 1231, 1171, 1106 cm⁻¹

30 NMR(¹H, 360MHz) : 0.88(t, J=7Hz, 3H) ; 0.98(d, J=7Hz, 3H) ; 1.28(8H) ; 1.40-1.80(7H) ; 2.27(t, J=7Hz, 2H) ; 2.62(m, 2H) ; 4.11(m, 2H) ; 7.10-7.30(5H) δ ppm.

NMR(¹³C) : 173.9(s) ; 142.7(s) ; 128.3(2d) ; 125.7(d) ; 62.6(t) ; 38.8(t) ; 35.5(t) ; 34.4(t) ; 33.3(t) ; 31.7(t) ; 29.6(d) ; 29.2(t) ; 29.0(t) ; 25.0(t) ; 22.6(t) ; 19.5(q) ; 14.1(q) δ ppm.

35 MS : 304(0, M⁺), 160(55), 131(30), 104(100), 91(56).

f. 3-methyl-5-phenylpentyl hexadecanoate

B. p. (bulb-to-bulb distillation) 250°/4 Pa

IR(CHCl₃) : 2927, 2855, 1727, 1456, 1235, 1178 cm⁻¹

NMR(¹H, 360MHz) : 0.88(t, J=7Hz, 3H) ; 0.97(d, J=7Hz, 3H) ; 1.26(24H) ; 1.48(2H) ; 1.55-1.80(5H) ; 2.27(t, J=7Hz, 2H) ; 2.62(m, 2H) ; 4.10(m, 2H) ; 7.15-7.30(5H) δ ppm.

5 NMR(¹³C) : 173.9(s) ; 142.6(s) ; 128.3(2d) ; 125.7(d) ; 62.6(t) ; 38.8(t) ; 35.5(t) ; 34.5(t) ; 33.3(t) ; 32.0(t) ; 29.7(2t) ; 29.7(d) ; 29.6(t) ; 29.5(t) ; 29.4(t) ; 29.3(t) ; 29.2(t) ; 25.1(t) ; 22.7(t) ; 19.5(q) ; 14.1(q) δ ppm.

MS : 416(0, M⁺), 160(43), 129(22), 115(36), 104(72), 91(68), 71(61), 57(100).

10 g. 7-p-menthanyl octanoate (cis/trans 70:30)

B. p. 105-115°/2.7 Pa

IR(CHCl₃) : 2929, 2858, 1724, 1453, 1232, 1172, 1106 cm⁻¹

NMR(¹H, 360MHz) : cis-7 : 0.86(d, J=7Hz, 6H) ; 0.88(t, J=7Hz, 6H) ; 2.30(t, J=7Hz, 2H) ; 4.02(d, J=7Hz, 2H) δ ppm.

15 trans-7 : 2.30(t, J=7Hz, 2H) ; 3.88(d, J=7Hz, 2H) δ ppm.

NMR(¹³C) : cis-7 : 174.0(s) ; 66.6(t) ; 43.0(d) ; 34.5(t) ; 33.9(d) ; 30.6(d) ; 29.9(t) ; 29.2(t) ; 29.1(t) ; 26.5(t) ; 25.6(t) ; 25.1(t) ; 20.3(q) ; 14.0(q) δ ppm.

20 trans-7 : 174.0(s) ; 69.5(t) ; 44.1(d) ; 37.5(d) ; 32.9(d) ; 31.7(t) ; 22.6(t) ; 19.8(q) ; 14.0(q) δ ppm.

MS : cis-7 : 282(0, M⁺), 138(23), 123(17), 109(35), 95(100), 81(27).

trans-7 : 282(0, M⁺), 138(27), 123(17), 109(22), 95(100), 81(35).

25 h. 9-decenyl octanoate

B. p. 120-121°/2.7 Pa

IR(CHCl₃) : 2830, 2857, 1725, 1466, 1171 cm⁻¹

NMR(¹H, 360MHz) : 0.88(t, J=7Hz, 3H) ; 1.30(18H) ; 1.61(4H) ; 2.04(broad q, J=7Hz, 2H) ; 2.29(t, J=7Hz, 2H) ; 4.06(t, J=7Hz, 2H) ; 4.93(broad d, J=11Hz, 1H) ; 4.99(broad d, J=17Hz, 1H) ; 5.80(m, 1H) δ ppm.

30 NMR(¹³C) : 173.9(s) ; 139.1(d) ; 114.2(t) ; 64.4(t) ; 34.5(t) ; 33.8(t) ; 31.8(t) ; 29.4(t) ; 29.3(t) ; 29.2(t) ; 29.1(t) ; 29.0(2t) ; 28.8(t) ; 26.0(t) ; 25.1(t) ; 22.7(t) ; 14.1(q) δ ppm.

MS : 282(0, M⁺), 145(38), 109(38), 96(86), 82(89), 68(91), 55(100).

35

i. 9-decenyl nonanoate

B. p. (bulb-to-bulb distillation) 130-160°/2.7 Pa

~~IR(CHCl₃): cm⁻¹~~

NMR(¹H, 360MHz): 0.88(t, J=7Hz, 3H); 1.20-1.45(20H); 1.61(4H); 2.04
(broad q, J=7Hz, 2H); 2.29(t, J=7Hz, 2H); 4.06(t, J=7Hz, 2H);
4.93(broad d, J=11Hz, 1H); 4.99(broad d, J=17Hz, 1H); 5.80(m, 1H)
δ ppm.

NMR(¹³C): 174.0(s); 139.1(d); 114.2(t); 64.4(t); 34.5(t); 33.8(t); 31.9(t);
29.4(t); 29.3(t); 29.2(2t); 29.1(t); 29.0(t); 28.7(t); 26.0(t); 25.1(t);
22.7(t); 14.1(q) δ ppm.

MS: 296(0, M⁺), 159(27), 138(28), 96(100), 82(75), 68(73), 55(83).

j. 9-decenyl decanoate

B. p. 144-145°/2.7 Pa

IR(CHCl₃): 2929, 2856 1726, 1466, 1178 cm⁻¹

NMR(¹H, 360MHz): 0.88(t, J=7Hz, 3H); 1.30(22H); 1.61(4H); 2.04(broad q,
J=7Hz, 2H); 2.29(t, J=7Hz, 2H); 4.05(t, J=7Hz, 2H); 4.93(broad d,
J=11Hz, 1H); 4.98(broad d, J=17Hz, 1H); 5.80(m, 1H) δ ppm.

NMR(¹³C): 173.9(s); 139.1(d); 114.2(t); 64.4(t); 34.5(t); 33.8(t); 31.9(t);
29.5(t); 29.3(t); 29.2(t); 29.1(t); 29.0(t); 28.7(t); 26.0(t); 25.1(t);
22.7(t); 14.1(q) δ ppm.

MS: 310(0, M⁺), 138(33), 109(29), 96(100), 82(82), 68(98).

k. 9-decenyl undecanoate

B. p. (bulb-to-bulb distillation) 130-150°/20 Pa

~~IR(CHCl₃): cm⁻¹~~

NMR(¹H, 360MHz): 0.88(t, J=7Hz, 3H); 1.20-1.45(24H); 1.61(4H);
2.04(broad q, J=7Hz, 2H); 2.29(t, J=7Hz, 2H); 4.06(t, J=7Hz, 2H);
4.93(broad d, J=11Hz, 1H); 4.99(broad d, J=17Hz, 1H); 5.81(m, 1H)
δ ppm.

NMR(¹³C): 173.9(s); 139.1(d); 114.2(t); 64.4(t); 34.5(t); 33.8(t); 32.0(t);
29.6(2t); 29.4(2t); 29.3(t); 29.1(t); 29.0(t); 28.8(t); 26.0(t); 25.1(t);
22.7(t); 14.1(q) δ ppm.

MS: 324(0, M⁺), 187(20), 138(43), 109(33), 96(100), 82(96), 68(89), 55(91).

l. 9-decenyl dodecanoate

B. p. 164-165°/4 Pa

IR(CHCl₃): 2928, 2856, 1726, 1216 cm⁻¹

NMR(¹H, 360MHz): 0.89(t, J=7Hz, 3H); 1.28(26H); 1.62(4H); 2.04(broad q,
J=7Hz, 2H); 2.29(t, J=7Hz, 2H); 4.06(t, J=7Hz, 2H); 4.93(broad d,
J=11Hz, 1H); 4.98(broad d, J=17Hz, 1H); 5.80(m, 1H) δ ppm.



NMR(^{13}C) : 174.0(s) ; 139.2(d) ; 114.2(t) ; 64.4(t) ; 34.5(t) ; 33.8(t) ; 32.0(t) ;
29.6(t) ; 29.5(t) ; 29.4(2t) ; 29.3(t) ; 29.2(t) ; 29.1(2t) ; 28.9(t) ; 28.7(t) ;
26.0(t) ; 25.1(t) ; 22.7(t) ; 14.1(q) δ ppm.

MS : 338(0, M^+), 138(48), 109(35), 96(100), 82(96), 68(89), 55(98).

5

m. 9-decenyl tridecanoate

B. p. (bulb-to-bulb distillation) 150-175 $^{\circ}$ /13 Pa

~~IR(CHCl_3) : cm^{-1}~~

10 NMR(^1H , 360MHz) : 0.88(t, $J=7\text{Hz}$, 3H) ; 1.20-1.45(28H) ; 1.61(4H) ;
2.04(broad q, $J=7\text{Hz}$, 2H) ; 2.29(t, $J=7\text{Hz}$, 2H) ; 4.05(t, $J=7\text{Hz}$, 2H) ;
4.93(broad d, $J=11\text{Hz}$, 1H) ; 4.99(broad d, $J=17\text{Hz}$, 1H) ; 5.81(m, 1H)
 δ ppm.

15 NMR(^{13}C) : 174.0(s) ; 139.1(d) ; 114.2(t) ; 64.4(t) ; 34.5(t) ; 33.8(t) ; 32.0(t) ;
29.7(t) ; 29.5(t) ; 29.4(t) ; 29.3(t) ; 29.2(t) ; 29.1(t) ; 29.0(t) ; 28.7(t) ;
26.0(t) ; 25.1(t) ; 22.7(t) ; 14.1(q) δ ppm.

MS : 352(0, M^+), 138(52), 110(41), 96(99), 82(83), 68(93), 55(100).

n. 9-decenyl tétradecanoate

B. p. (bulb-to-bulb distillation) 200-220 $^{\circ}$ /4 Pa

20 ~~IR(CHCl_3) : cm^{-1}~~

NMR(^1H , 360MHz) : 0.88(t, $J=7\text{Hz}$, 3H) ; 1.28(30H) ; 1.62(4H) ; 2.04(broad q,
 $J=7\text{Hz}$, 2H) ; 2.29(t, $J=7\text{Hz}$, 2H) ; 4.06(t, $J=7\text{Hz}$, 2H) ; 4.93(broad d,
 $J=11\text{Hz}$, 1H) ; 4.99(broad d, $J=17\text{Hz}$, 1H) ; 5.81(m, 1H) δ ppm.

25 NMR(^{13}C) : 174.0(s) ; 139.1(d) ; 114.2(t) ; 64.4(t) ; 34.5(t) ; 34.5(t) ; 33.8(t) ;
32.0(t) ; 29.7(t) ; 29.5(t) ; 29.4(t) ; 29.3(2t) ; 29.1(t) ; 29.0(t) ; 28.7(t) ;
26.0(t) ; 25.1(t) ; 22.7(t) ; 14.1(q) δ ppm.

MS : 366(0, M^+), 138(52), 110(47), 96(100), 82(96), 55(96).

o. 9-decenyl pentadecanoate

30 B. p. (bulb-to-bulb distillation) 175-210 $^{\circ}$ /13 Pa

~~IR(CHCl_3) : cm^{-1}~~

35 NMR(^1H , 360MHz) : 0.88(t, $J=7\text{Hz}$, 3H) ; 1.20-1.45(32H) ; 1.62(4H) ;
2.04(broad q, $J=7\text{Hz}$, 2H) ; 2.29(t, $J=7\text{Hz}$, 2H) ; 4.06(t, $J=7\text{Hz}$, 2H) ;
4.93(broad d, $J=11\text{Hz}$, 1H) ; 4.99(broad d, $J=17\text{Hz}$, 1H) ; 5.81(m, 1H)
 δ ppm.

NMR(^{13}C) : 174.0(s) ; 139.1(d) ; 114.2(t) ; 64.4(t) ; 34.5(t) ; 33.8(t) ; 32.0(t) ;
29.7(2t) ; 29.5(t) ; 29.4(t) ; 29.3(t) ; 29.2(t) ; 29.1(t) ; 29.0(t) ; 28.7(t) ;
26.0(t) ; 25.1(t) ; 22.7(t) ; 14.1(q) δ ppm.

MS : 380(0, M^+), 138(50), 110(30), 96(100), 82(85), 68(79), 55(80).



p. 9-decenyl hexadecanoate

NMR(^1H , 360MHz) : 5.84(m, 1H) ; 4.95(m, 2H) ; 4.05(t, 2H) ; 3.73(q, 4H) ;
2.31(t, 2H) ; 2.05(broad q, 2H) ; 1.61(m, 4H) ; 1.2-1.4(broad m, 30H) ;
0.89(m, 3H) δ ppm.

5 NMR(^{13}C) : 14.12(q) ; 18.44(q) ; 22.72(t) ; 25.06(t) ; 25.95(t) ; 28.68(t) ;
28.92(t) ; 29.05(t) ; 29.21(t) ; 29.30(t) ; 29.39(t) ; 29.51(t) ; 29.64(t) ;
29.71(t) ; 31.95(t) ; 33.80(t) ; 34.45(t) ; 58.45(t) ; 64.41(t) ; 114.17(t) ;
139.16(d) ; 174.07(s) δ ppm.

MS : 394(0, M^+), 96(72), 83(70), 82(88), 55(100).

10

q. 1-methylpentyl hexadecanoate

NMR(^1H , 360MHz) : 4.90(m, 1H) ; 2.26(t, 2H) ; 1.65(m, 4H) ; 1.25-1.32(m,
28H) ; 1.20(d, 3H) ; 0.89(q, 6H) δ ppm.

15 NMR(^{13}C) : 13.97(q) ; 14.00(q) ; 20.40(q) ; 22.56(t) ; 22.7(t) ; 25.1(t) ; 27.6(t) ;
29.2(t) ; 29.3(t) ; 29.4(t) ; 29.5(t) ; 29.6(t) ; 29.7(t) ; 29.73(t) ; 31.97(t) ;
34.83(t) ; 35.75(t) ; 70.74(d) ; 173.50(s) δ ppm.

MS : 84(100), 69(50), 57(70), 55(62), 43(98).

r. 7-p-menthanyl hexadecanoate

20 NMR(^1H , 360MHz) : 0.80-0.95(m, 9H) ; 1.2-2.0(broad m, 37H) ; 2.32(t, 2H) ;
3.88 et 4.02(d, 2H) δ ppm.

NMR(^{13}C) : 14.09(t) ; 19.8(t) ; 20.2(t) ; 22.7(t) ; 25.12(t) ; 22.56(t) ; 26.44(t) ;
29.10(t) ; 29.22(t) ; 29.30(t) ; 29.38(t) ; 29.51(t) ; 29.63(t) ; 29.71(t) ;
29.92(t) ; 30.54(d) ; 31.97(t) ; 32.90(d) ; 33.90(d) ; 34.50(t) ; 37.50(d) ;
25 42.98(d) ; 44.11(d) ; 66.62(t) ; 69.50(t) ; 174.05(s) δ ppm.

MS : 93(70), 77(33), 69(100), 43(48), 41(92).

s. hexyl hexadecanoate

30 NMR(^1H , 360MHz) : 4.02(t, 2H) ; 3.86(broad q, 2H) ; 2.29(t, 2H) ;
1.60(broad m, 4H) ; 1.2-1.4(broad m, 28H) ; 0.90(m, 6H) δ ppm.

NMR(^{13}C) : 14.1(q) ; 13.97(q) ; 22.58(t) ; 22.74(t) ; 25.11(t) ; 25.68(t) ; 28.75(t) ;
29.24(t) ; 29.34(t) ; 29.41(t) ; 29.54(t) ; 29.67(t) ; 29.72(t) ; 29.74(t) ;
31.51(t) ; 32.00(t) ; 34.50(t) ; 58.42(t) ; 64.46(t) ; 174.07(s) δ ppm.

MS : 340(2, M^+), 84(100), 57(55), 56(58), 43(80).

35

t. (Z)-3-hexenyl hexadecanoate

NMR(^1H , 360MHz) : 5.45(m, 1H) ; 5.26(m, 1H) ; 4(t, 2H) ; 3.42(s, 4H) ; 2.2-3.3(m, 8H) ; 2.0(m, 1H) ; 1.6(m, 8H) ; 1.2(broad m, 30H) ; 0.8-1.0(m, 6H) δ ppm.

5 NMR(^{13}C) : 14.14(q) ; 14.24(q) ; 20.63(t) ; 22.73(t) ; 25.02(t) ; 25.59(t) ; 26.37(t) ; 26.72(t) ; 26.82(t) ; 29.20(t) ; 29.32(t) ; 29.40(t) ; 29.51(t) ; 29.73(t) ; 31.97(t) ; 34.39(t) ; 93.77(t) ; 123.83(d) ; 134.50(d) ; 173.93(s) δ ppm.

MS : 83(25), 82(100), 67(38), 55(27), 43(14).

10

u. (E)-3,7-dimethyl-2,6 octadienyl hexadecanoate

NMR(^1H , 360MHz) : 0.87(t, 3H) ; 1.26(m, 26H) ; 1.6(s, 3H) ; 1.68(s, 3H) ; 1.70(s, 3H) ; 2.0-2.15(m, 4H) ; 2.30(t, 2H) ; 4.6(d, 2H) ; 5.09(t, 1H) ; 5.35(t, 1H) δ ppm.

15 NMR(^{13}C) : 14.12(q) ; 16.48(q) ; 17.69(q) ; 27.73(t) ; 25.08(t) ; 25.68(t) ; 26.38(t) ; 29.22(t) ; 29.33(t) ; 29.41(t) ; 29.53(t) ; 29.67(t) ; 29.72(t) ; 31.99(t) ; 34.45(t) ; 35.59(t) ; 61.19(t) ; 116.60(d) ; 123.85(d) ; 131.80(s) ; 142.06(s) ; 173.91(s) δ ppm.

MS : 392(0, M^+), 93(94), 69(100), 43(89), 41(70).

20

Example 2Preparation ^{of} di-esters

25

General method

The same method as that described in Example 1 was used, but employing half the molar amounts of the corresponding diacyl dichloride
30 $[(\text{CH}_2)_n[\text{C}(\text{O})\text{Cl}]_2, n=0 \text{ to } 6]$.

The following di-esters were prepared according to this general method :

a. bis(9-decenyl) oxalate

35

B. p. (bulb-to-bulb distillation): 200°/40 Pa

IR(CHCl_3) : 2930, 2860, 1770, 1740, 1460, 1312, 1175, 912 cm^{-1}

NMR(^1H , 360MHz) : 1.25-1.45(20H) ; 1.74(m, 4H) ; 2.04(broad q, $J=7\text{Hz}$, 4H) ; 4.28(t, $J=7\text{Hz}$, 4H) ; 4.93(broad d, $J=11\text{Hz}$, 2H) ; 4.99(broad d, $J=17\text{Hz}$, 2H) ; 5.80(m, 2H) δ ppm.



NMR(^{13}C) : 158.1(s) ; 139.0(d) ; 114.2(t) ; 67.1(t) ; 33.8(t) ; 29.3(t) ; 29.1(t) ;
29.0(t) ; 28.9(t) ; 28.3(t) ; 25.7(t) δ ppm.

MS : 366(0, M^+), 138(9), 109(13), 96(27), 83(55), 69(45), 55(100).

5 b. bis(9-decenyl) malonate

B. p. (bulb-to-bulb distillation): 210°/40 Pa

IR(CHCl_3) : 2940, 2862, 1740, 1465, 1336, 1276, 1155, 915 cm^{-1}

NMR(^1H , 360MHz) : 1.25-1.45(20H) ; 1.64(m, 4H) ; 2.04(broad q, $J=7\text{Hz}$, 4H) ; 3.37(s, 2H) ; 4.14(t, $J=7\text{Hz}$, 4H) ; 4.93(broad d, $J=11\text{Hz}$, 2H) ; 4.99
10 (broad d, $J=17\text{Hz}$, 2H) ; 5.81(m, 2H) δ ppm.

NMR(^{13}C) : 166.6(s) ; 139.1(d) ; 114.2(t) ; 65.6(t) ; 41.7(t) ; 33.8(t) ; 29.4(t) ;
29.2(t) ; 29.1(t) ; 28.9(t) ; 28.5(t) ; 25.8(t) δ ppm.

MS : 380(0, M^+), 138(25), 109(21), 105(42), 96(60), 83(100), 68(65), 55(95).

15 c. bis(9-decenyl) butanedioate

IR(CHCl_3) : 2935, 2860, 1735, 1460, 1160, 995, 910 cm^{-1}

NMR(^1H , 360MHz) : 1.25-1.45(20H) ; 1.63(m, 4H) ; 2.04(broad q, $J=7\text{Hz}$, 4H) ; 2.62(s, 4H) ; 4.08(t, $J=7\text{Hz}$, 4H) ; 4.93(broad d, $J=11\text{Hz}$, 2H) ;
4.99(broad d, $J=17\text{Hz}$, 2H) ; 5.81(m, 2H) δ ppm.

NMR(^{13}C) : 172.3(s) ; 139.1(d) ; 114.2(t) ; 64.9(t) ; 33.8(t) ; 29.4(t) ; 29.3(t) ;
20 29.2(t) ; 29.1(t) ; 28.9(t) ; 28.7(t) ; 25.9(t) δ ppm.

MS : 394(0, M^+), 138(10), 119(22), 101(60), 97(38), 83(100), 69(44), 55(76).

25 d. bis(9-decenyl) pentanedioate

IR(CHCl_3) : 2940, 2880, 1740, 1464, 1180, 1000, 918 cm^{-1}

NMR(^1H , 360MHz) : 1.25-1.45(20H) ; 1.62(m, 4H) ; 1.95(t, $J=7$, 7Hz, 2H) ;
2.04(broad q, $J=7\text{Hz}$, 4H) ; 2.37(t, $J=7\text{Hz}$, 4H) ; 4.06(t, $J=7\text{Hz}$, 4H) ;
4.93(broad d, $J=11\text{Hz}$, 2H) ; 4.99(broad d, $J=17\text{Hz}$, 2H) ; 5.81(m, 2H)
30 δ ppm.

NMR(^{13}C) : 173.0(s) ; 139.1(d) ; 114.2(t) ; 64.6(t) ; 33.8(t) ; 33.4(t) ; 29.4(t) ;
29.2(t) ; 29.1(t) ; 28.9(t) ; 28.7(t) ; 25.9(t) δ ppm.

MS : 408(0, M^+), 115(100), 97(14), 87(20), 83(26), 69(19), 55(39).

35 e. bis(9-decenyl) hexanedioate

IR(CHCl_3) : 2942, 2864, 1740, 1466, 1180, 1000, 918 cm^{-1}

NMR(^1H , 360MHz) : 1.25-1.45(20H) ; 1.55-1.75(8H) ; 2.04(broad q, $J=7\text{Hz}$, 4H) ; 2.32(m, 4H) ; 4.06(t, $J=7\text{Hz}$, 4H) ; 4.93(broad d, $J=11\text{Hz}$, 2H) ;
4.99(broad d, $J=17\text{Hz}$, 2H) ; 5.81(m, 2H) δ ppm.

NMR(^{13}C) : 173.4(s) ; 139.1(d) ; 114.2(t) ; 64.5(t) ; 34.0(t) ; 33.8(t) ; 29.4(t) ;
29.2(t) ; 29.0(t) ; 28.9(t) ; 28.7(t) ; 25.9(t) ; 24.5(t) δ ppm.

MS : 422(0, M^+), 129(90), 111(64), 101(31), 95(23), 83(60), 67(41), 55(100).

5 f. (E,E)-bis(3,7-dimethyl-2,6-octadienyl) oxalate

IR(CHCl_3) : 2929, 1740, 1449, 1378, 1302, 1168 cm^{-1}

NMR(^1H , 360MHz) : 1.60(s, 6H) ; 1.68(s, 6H) ; 1.75(s, 6H) ; 2.09(8H) ; 4.80(d,
 $J=7\text{Hz}$, 4H) ; 5.07(m, 2H) ; 5.41(broad t, $J=7\text{Hz}$, 2H) δ ppm.

10 NMR(^{13}C) : 158.0(s) ; 144.3(s) ; 132.0(s) ; 123.6(d) ; 116.9(d) ; 63.8(t) ; 39.6(t) ;
26.2(t) ; 25.7(q) ; 17.7(q) ; 16.6(q) δ ppm.

MS : 362(0, M^+), 135(7), 93(20), 81(29), 69(100).

g. (E,E)-bis(3,7-dimethyl-2,6-octadienyl) malonate

IR(CHCl_3) : 2930, 1728, 1447, 1379, 1278, 1149, 983 cm^{-1}

15 NMR(^1H , 360MHz) : 1.60(s, 6H) ; 1.69(s, 6H) ; 1.71(s, 6H) ; 2.08(8H) ; 3.38(s,
2H) ; 4.65(d, $J=7\text{Hz}$, 4H) ; 5.08(m, 2H) ; 5.34(broad t, $J=7\text{Hz}$, 2H)
 δ ppm.

NMR(^{13}C) : 166.6(s) ; 142.9(s) ; 131.9(s) ; 123.7(d) ; 117.8(d) ; 62.4(t) ; 41.7(t) ;
39.6(t) ; 26.4(t) ; 25.7(q) ; 17.7(q) ; 16.5(q) δ ppm.

20 MS : 376(0, M^+), 136(17), 121(15), 93(39), 81(33), 69(100).

h. (E,E)-bis(3,7-dimethyl-2,6-octadienyl) butanedioate

IR(CHCl_3) : 2930, 1729, 1446, 1384, 1231, 1162 cm^{-1}

25 NMR(^1H , 360MHz) : 1.61(s, 6H) ; 1.69(s, 6H) ; 1.70(s, 6H) ; 2.08(8H) ; 2.64(s,
4H) ; 4.62(d, $J=7\text{Hz}$, 4H) ; 5.08(m, 2H) ; 5.34(broad t, $J=7\text{Hz}$, 2H)
 δ ppm.

NMR(^{13}C) : 172.3(s) ; 142.3(s) ; 131.8(s) ; 123.8(d) ; 118.3(d) ; 61.7(t) ; 39.6(t) ;
29.3(t) ; 26.4(t) ; 25.7(q) ; 17.7(q) ; 16.5(q) δ ppm.

30 MS : 390(0, M^+), 136(17), 121(19), 93(62), 81(27), 69(100).

i. (E,E)-bis(3,7-dimethyl-2,6-octadienyl) pentanedioate

IR(CHCl_3) : 2930, 1727, 1450, 1232, 1176 cm^{-1}

35 NMR(^1H , 360MHz) : 1.61(s, 6H) ; 1.68(s, 6H) ; 1.70(s, 6H) ; 1.96(m, 2H) ;
2.07(8H) ; 2.37(t, $J=7\text{Hz}$, 4H) ; 4.59(d, $J=7\text{Hz}$, 4H) ; 5.08(m, 2H) ;
5.33(broad t, $J=7\text{Hz}$, 2H) δ ppm.

NMR(^{13}C) : 172.9(s) ; 142.2(s) ; 131.8(s) ; 123.8(d) ; 118.4(d) ; 61.4(t) ; 39.6(t) ;
33.4(t) ; 26.3(t) ; 25.7(q) ; 20.3(t) ; 17.7(q) ; 16.5(q) δ ppm.

MS : 404(0, M^+), 136(20), 121(18), 93(55), 81(48), 69(100).

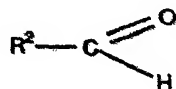
j. (E,E)-bis(3,7-dimethyl-2,6-octadienyl) hexanedioateIR(CHCl₃) : 2931, 1727, 1446, 1384, 1233, 1174 cm⁻¹

NMR(¹H, 360MHz) : 1.60(s, 6H) ; 1.68(4H) ; 1.68(s, 6H) ; 1.70(6H) ;
2.07(8H) ; 2.33(4H) ; 4.59(d, J=7Hz, 4H) ; 5.08(m, 2H) ; 5.33(broad t,
J=7Hz, 2H) δ ppm.

NMR(¹³C) : 173.3(s) ; 142.2(s) ; 131.8(s) ; 123.8(d) ; 118.4(d) ; 61.3(t) ; 39.8(t) ;
34.0(t) ; 26.4(t) ; 25.7(q) ; 24.5(t) ; 17.7(q) ; 16.5(q) δ ppm.

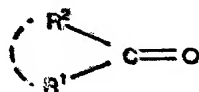
MS : 418(0, M⁺), 135(15), 121(15), 93(52), 81(32), 69(100).Example 3Preparation of enol-esters15 General method

A 500 ml three-neck flask under argon was charged with 0.162 mole of the appropriate aldehyde of formula



2.5 g (26 mmole) of anhydrous potassium acetate, 34.45 g (0.34 mole) of triethylamine and 250 ml of acetic anhydride. After heating for 6 h at 120°, the reaction mixture was cooled to room temperature, poured on ice, extracted three times with petroleum ether 30-50°, washed 6 times with 100 ml of saturated NaHCO₃, then with water to neutrality. After drying over Na₂SO₄, filtering, concentrating under vacuum and distilling on a Vigreux column, there was obtained the enolacetate corresponding to the starting aldehyde, in the form of a mixture of E and Z isomers. The latter

When starting from a ketone of formula



there was followed a method analogous to that described by T. Taapken et al., J. Chem. Soc. Perkin Trans. I, 1994, 1439, wherein the ketone is dissolved

in the acetic anhydride and treated with p-toluenesulfonic acid as catalyst, and the formed acetic acid is eliminated to obtain the corresponding enolacetate.

The enolacetates thus prepared are then converted into the enol-esters of formula (I) proceeding as follow.

A 500 ml three-neck flask under argon was charged with 0.05 mole of enolacetate and 75 ml of absolute tetrahydrofuran. The mixture was cooled to -60/-70° (dry ice/acetone bath) and a solution of 6.16 g (0.055 mole) of t-BuOK in 60 ml of absolute THF was introduced dropwise (slightly exothermic, the reaction mixture becomes strongly yellow). Stirring was kept for 1 h at -70° and 0.055 mole of the appropriate acyl chloride, in solution in 25 ml of absolute THF, were added dropwise. After 2 h at -70°, the cooling bath was removed and 60 ml of a saturated solution of NaHCO₃ were quickly added (the temperature rises quickly to -10°). The mixture was extracted twice with sulfuric ether, washed once with saturated NaHCO₃, once with water saturated with salt, dried (Na₂SO₄) and concentrated under vacuum. The product is then purified by flash-chromatography (column diameter = 9 cm, hexane/ether 98:2) and, after combining the useful layers, concentrated to dry under absolute vacuum.

According to this method, there were prepared the following enol-esters :

a. 2-(4-tert-butylbenzyl)-1-propenyl acetate

Isomer (Z)

NMR(¹H, 360MHz, CDCl₃) : 1.32(s, 9H) ; 1.59(s, 3H) ; 2.16(s, 3H) ; 3.43(s, 2H) ; 6.99(broad s, 1H) ; 7.10(d, J=8, 2H) ; 7.31(d, J=8, 2H) δ ppm.

NMR(¹³C, 90MHz, CDCl₃) : 17.4(q) ; 20.8(q) ; 31.4(q) ; 31.4(q) ; 31.4(q) ; 35.3(t) ; 125.3(d) ; 125.3(d) ; 128.3(d) ; 128.3(d) ; 130.5(d) ; 34.4(s) ; 121.2(s) ; 136.0(s) ; 149.0(s) ; 168.3(s) δ ppm.

MS : 246(13, M⁺); m/e, 204(17), 189(73), 171(7), 159(8), 147(31), 129(33), 119(100), 105(9), 91(40), 77(8), 71(7), 57(60), 43(64).

Isomer (E)

NMR(¹H, 360MHz, CDCl₃) : 1.32(s, 9H) ; 1.62(s, 3H) ; 2.15(s, 3H) ; 3.24(s, 2H) ; 7.05(broad s, 1H) ; 7.12(d, J=8, 2H) ; 7.31(d, J=8, 2H) δ ppm.

NMR(¹³C, 90MHz, CDCl₃) : 13.6(q) ; 20.8(q) ; 31.4(q) ; 31.4(q) ; 31.4(q) ; 39.8(t) ; 125.3(d) ; 125.3(d) ; 128.4(d) ; 128.4(d) ; 131.2(d) ; 34.4(s) ; 121.4(s) ; 135.9(s) ; 149.2(s) ; 168.3(s) δ ppm.

MS : 246(12, M⁺); m/e, 204(41), 189(100), 171(3), 159(7), 147(42), 131(21), 119(57), 105(9), 91(32), 77(6), 71(8), 57(45), 43(58).

b. 3,7-dimethyl-1,6-octadienyl acetate

The analytical characteristics of this compound were identical to those published in the literature (see, D.P. Simmons et al., ref. cited).

5 c. 3,7-dimethyl-1,6-octadienyl octanoate

Isomer (Z)

NMR(^1H , 360MHz, CDCl_3): 0.89(t, J=7, 3H); 0.99(d, J=7, 3H); 1.30(m, 10H); 1.59(s, 3H); 1.68(s, 3H); 1.94(m, 2H); 2.39(t, J=7, 2H); 2.68(m, 1H); 4.67(dd, $J_1=6$, $J_2=10$, 1H); 5.10(m, 1H); 7.00(d, J=6, 1H) δ ppm.

10 NMR(^{13}C , 90MHz, CDCl_3): 14.1(q); 17.6(q); 20.9(q); 25.7(q); 22.6(t); 24.8(t); 25.9(t); 28.9(t); 29.0(t); 31.7(t); 34.2(t); 37.4(t); 29.4(d); 120.1(d); 124.5(d); 133.1(d); 131.3(s); 171.0(s) δ ppm.

MS: 280(0, M^+); m/e, 198(1), 182(2), 154(8), 136(35), 127(100), 121(30), 109(20), 93(12), 82(17), 69(23), 57(96), 41(32).

15

Isomer (E)

NMR(^1H , 360MHz, CDCl_3): 0.89(t, J=7, 3H); 1.02(d, J=7, 3H); 1.30(m, 10H); 1.59(s, 3H); 1.68(s, 3H); 1.96(m, 2H); 2.15(m, 1H); 2.36(t, J=7, 2H); 5.07(m, 1H); 5.29(dd, $J_1=8$, $J_2=12$, 1H); 7.08(d, J=12, 1H) δ ppm.

20 NMR(^{13}C , 90MHz, CDCl_3): 14.1(q); 17.7(q); 20.9(q); 25.7(q); 22.6(t); 24.7(t); 25.7(t); 28.9(t); 29.1(t); 31.6(t); 34.1(t); 37.2(t); 32.0(d); 120.4(d); 124.3(d); 134.6(d); 131.5(s); 171.2(s) δ ppm.

MS: 280(0, M^+); m/e, 198(1), 185(1), 154(7), 136(32), 127(93), 121(26), 109(19), 93(11), 82(15), 69(23), 57(100), 41(32).

25

d. 2-methyl-1-undecenyl acetate

Isomer (Z)

NMR(^1H , 360MHz, CDCl_3): 0.88(t, J=7, 3H); 1.27(m, 12H); 1.39(m, 2H); 1.63(split s, 3H); 2.11(m, 2H); 2.13(s, 3H); 6.83(broad s, 1H) δ ppm.

30 NMR(^{13}C , 90MHz, CDCl_3): 14.1(q); 17.4(q); 20.7(q); 22.7(t); 27.0(t); 29.3-29.6(t); 29.3-29.6(t); 29.3-29.6(t); 29.3-29.6(t); 29.3-29.6(t); 31.9(t); 120.0(d); 122.3(s); 168.3(s) δ ppm.

MS: 226(3, M^+); m/e, 184(33), 166(1), 141(2), 124(3), 110(6), 95(14), 82(15), 71(100), 57(13), 43(50).

35

Isomer (E)

NMR(^1H , 360MHz, CDCl_3): 0.88(t, J=7, 3H); 1.27(m, 12H); 1.40(m, 2H); 1.66(split s, 3H); 1.95(m, 2H); 2.13(s, 3H); 6.88(broad s, 1H) δ ppm.

NMR(^{13}C , 90MHz, CDCl_3) : 13.6(q) ; 14.1(q) ; 20.8(q) ; 22.7(t) ; 27.6(t) ;
29.2(t) ; 29.3(t) ; 29.5(t) ; 29.6(t) ; 31.9(t) ; 33.9(t) ; 130.2(d) ; 122.0(s) ;
168.3(s) δ ppm.

MS : 226(2, M^+); m/e, 184(25), 166(1), 141(2), 123(3), 110(5), 95(12), 81(18),
71(100), 58(9), 43(40).

e. 2-methyl-1-undecenyl octanoate

Isomer (Z)

NMR(^1H , 360MHz, CDCl_3) : 0.88(t, J=7, 6H) ; 1.27(m, 22H) ; 1.39(m, 2H) ;
1.52(split s, 3H) ; 2.10(t, J=7, 2H) ; 2.37(t, J=7, 2H) ; 6.85(broad s, 1H)
 δ ppm.

NMR(^{13}C , 90MHz, CDCl_3) : 14.0(q) ; 14.1(q) ; 17.4(q) ; 22.6(t) ; 22.7(t) ;
24.9(t) ; 27.1(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ;
28.9-29.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ; 31.7(t) ; 31.9(t) ; 34.2(t) ;
129.9(d) ; 122.2(s) ; 171.1(s) δ ppm.

MS : 310(0, M^+); m/e, 184(10), 127(85), 109(10), 95(6), 81(10), 71(25), 57(100),
43(10).

Isomer (E)

NMR(^1H , 360MHz, CDCl_3) : 0.88(t, J=7, 6H) ; 1.27(m, 22H) ; 1.40(m, 2H) ;
1.67(split s, 3H) ; 1.95(t, J=7, 2H) ; 2.39(t, J=7, 2H) ; 6.89(broad s, 1H)
 δ ppm.

NMR(^{13}C , 90MHz, CDCl_3) : 13.6(q) ; 14.0(q) ; 14.1(q) ; 22.6(t) ; 22.7(t) ;
24.9(t) ; 27.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ; 28.9-29.6(t) ;
28.9-29.6(t) ; 28.9-29.6(t) ; 31.7(t) ; 31.9(t) ; 34.0(t) ; 34.2(t) ; 130.1(d) ;
121.9(s) ; 171.1(s) δ ppm.

MS : 310(0, M^+); m/e, 184(10), 127(87), 109(10), 97(7), 81(13), 71(28), 57(100),
43(9).

f. 1-undecenyl acetate

Isomer (Z)

NMR(^1H , 360MHz, CDCl_3) : 0.88(t, J=7, 3H) ; 1.28(m, 12H) ; 1.37(m, 2H) ;
2.12(m, 2H) ; 2.14(s, 3H) ; 4.87(dt, $J_1=J_2=6$, 1H) ; 6.99(d, J=6, 1H)
 δ ppm.

NMR(^{13}C , 90MHz, CDCl_3) : 14.1(q) ; 20.8(q) ; 22.7(t) ; 24.4(t) ; 29.2-29.6(t) ;
29.2-29.6(t) ; 29.2-29.6(t) ; 29.2-29.6(t) ; 29.2-29.6(t) ; 31.9(t) ; 114.4(d) ;
134.0(d) ; 168.2(s) δ ppm.

MS : 212(0, M⁺); m/e, 170(1), 152(3), 124(5), 110(6), 96(26), 82(34), 68(18), 57(33), 43(100).

Isomer (E)

5 NMR(¹H, 360MHz, CDCl₃) : 0.88(t, J=7, 3H) ; 1.27(m, 12H) ; 1.37(m, 2H) ; 1.99(q, J=7, 2H) ; 2.11(s, 3H) ; 5.41(dt, J₁=7, J₂=12, 1H) ; 7.06(d, J=12, 1H) δ ppm.

NMR(¹³C, 90MHz, CDCl₃) : 14.1(q) ; 20.7(q) ; 22.7(t) ; 27.3(t) ; 29.1-29.6(t) ; 29.1-29.6(t) ; 29.1-29.6(t) ; 29.1-29.6(t) ; 31.9(t) ; 115.1(d) ; 135.4(d) ; 168.3(s) δ ppm.

10 MS : 212(0, M⁺); m/e, 170(1), 152(4), 124(7), 110(8), 96(34), 82(41), 68(19), 57(39), 43(100).

g. 3-methyl-5-phenyl-1-pentenyl acetate

15 Isomer (Z)

NMR(¹H, 360MHz, CDCl₃) : 1.02(d, J=7, 3H) ; 1.54(m, 1H) ; 1.69(m, 1H) ; 2.10(s, 3H) ; 2.48-2.78(m, 3H) ; 4.72(dd, J₁=6, J₂=10, 1H) ; 7.03(d, J=6, 1H) ; 7.17(m, 3H) ; 7.26(m, 2H) δ ppm.

20 NMR(¹³C, 90MHz, CDCl₃) : 20.7(q) ; 21.0(q) ; 33.7(t) ; 38.9(t) ; 29.2(d) ; 119.8(d) ; 125.6(d) ; 128.2(d) ; 128.2(d) ; 128.4(d) ; 128.4(d) ; 133.4(d) ; 142.5(s) ; 168.1(s) δ ppm.

MS : 218(1, M⁺); m/e, 176(3), 158(39), 143(27), 131(10), 117(6), 104(16), 91(45), 71(43), 65(13), 51(7), 43(100).

25 Isomer (E)

NMR(¹H, 360MHz, CDCl₃) : 1.06(d, J=7, 3H) ; 1.62(m, 2H) ; 2.12(s, 3H) ; 2.18(m, 1H) ; 2.60(m, 2H) ; 5.33(dd, J₁=9, J₂=13, 1H) ; 7.09(d, J=13, 1H) ; 7.17(m, 3H) ; 7.27(m, 2H) δ ppm.

30 NMR(¹³C, 90MHz, CDCl₃) : 20.7(q) ; 21.0(q) ; 33.5(t) ; 38.8(t) ; 32.1(d) ; 120.3(d) ; 125.7(d) ; 128.3(d) ; 128.3(d) ; 128.4(d) ; 128.4(d) ; 134.9(d) ; 142.3(s) ; 168.3(s) δ ppm.

MS : 218(1, M⁺); m/e, 176(4), 158(34), 143(25), 131(9), 117(5), 104(16), 91(44), 71(48), 65(12), 51(6), 43(100).

35 h. 3-methyl-5-phenyl-1-pentenyl octanoate

Isomer (Z)

NMR(¹H, 360MHz, CDCl₃) : 0.89(t, J=7, 3H) ; 1.02(d, J=7, 3H) ; 1.30(m, 8H) ; 1.49-1.76(m, 4H) ; 2.36(t, J=7, 2H) ; 2.48-2.78(m, 3H) ; 4.72(dd, J₁=7, J₂=10, 1H) ; 7.05(d, J=7, 1H) ; 7.17(m, 3H) ; 7.26(m, 2H) δ ppm.

NMR(^{13}C , 90MHz, CDCl_3): 14.1(q); 21.0(q); 22.6(t); 24.7(t); 28.9(t); 29.0(t); 31.7(t); 33.7(t); 34.1(t); 38.9(t); 29.3(d); 119.7(d); 125.6(d); 128.2(d); 128.2(d); 128.4(d); 128.4(d); 133.4(d); 142.5(s); 171.0(s) δ ppm.

5 MS: 308(0, M^+); m/e, 176(8), 158(78), 143(18), 127(100), 104(36), 91(51), 71(10), 57(71), 43(14).

Isomer (E)

10 NMR(^1H , 360MHz, CDCl_3): 0.89(t, J=7, 3H); 1.07(d, J=7, 3H); 1.30(m, 8H); 1.65(m, 4H); 2.18(m, 1H); 2.37(t, J=7, 2H); 2.60(m, 2H); 5.33(dd, $J_1=8$, $J_2=12$, 1H); 7.10(d, J=12, 1H); 7.17(m, 3H); 7.27(m, 2H) δ ppm.

15 NMR(^{13}C , 90MHz, CDCl_3): 14.1(q); 21.0(q); 22.6(t); 24.7(t); 28.9(t); 29.0(t); 31.6(t); 33.6(t); 34.1(t); 38.9(t); 32.1(d); 120.1(d); 125.7(d); 128.3(d); 128.3(d); 128.4(d); 128.4(d); 135.0(d); 142.54(s); 171.1(s) δ ppm.

MS: 308(0, M^+); m/e, 198(1), 176(10), 158(66), 143(20), 127(95), 104(34), 91(56), 71(13), 57(100), 43(20).

20 i. methyl 3-acetoxy-2-pentyl-2-cyclopentene-1-acetate

NMR(^1H , 360MHz, CDCl_3): 0.88(t, J=7, 3H); 1.28(m, 5H); 1.41(m, 1H); 1.61(m, 1H); 1.79(m, 1H); 2.14(s, 3H); 2.03-2.26(m, 3H); 2.46(m, 2H); 2.56(dd, $J_1=4$, $J_2=15$, 1H); 3.04(m, 1H); 3.68(s, 3H) δ ppm.

25 NMR(^{13}C , 90MHz, CDCl_3): 14.0(q); 20.7(q); 51.5(q); 22.4(t); 24.5(t); 26.8(t); 27.2(t); 29.6(t); 31.7(t); 38.6(t); 39.7(d); 128.3(s); 145.4(s); 168.5(s); 173.2(s) δ ppm.

MS: 268(1, M^+); m/e, 226(10), 208(2), 195(3), 169(3), 153(100), 137(4), 123(3), 109(10), 97(35), 83(10), 67(9), 55(8), 43(31).

30 j. methyl 3-octanoyloxy-2-pentyl-1-cyclopentene-1-acetate

NMR(^1H , 360MHz, CDCl_3): 0.88(t, J=7, 3H); 0.89(t, J=7, 3H); 1.30(m, 14H); 1.53-1.72(m, 3H); 1.79(m, 1H); 2.08(m, 1H); 2.19(m, 2H); 2.31-2.51(m, 4H); 2.57(dd, $J_1=4$, $J_2=15$, 1H); 3.05(m, 1H); 3.68(s, 3H) δ ppm.

35 NMR(^{13}C , 90MHz, CDCl_3): 14.0(q); 14.1(q); 51.5(q); 22.4(t); 22.6(t); 24.5(t); 25.0(t); 26.8(t); 27.1(t); 28.9(t); 29.1(t); 29.6(t); 31.7(t); 31.7(t); 34.2(t); 38.6(t); 39.6(d); 128.2(s); 145.3(s); 171.5(s); 173.3(s) δ ppm.

MS : 352(0, M⁺); m/e, 279(2), 226(92), 208(3), 194(4), 169(4), 153(100),
127(12), 109(7), 97(6), 81(5), 67(5), 57(22), 43(10).

5

Example 4

Test on fabrics

Several tests were carried out on fabrics, under a variety of conditions, said
10 fabrics having been treated according to the following general method.

General method of treatment of textiles

A standard cotton swatch of 32 g was placed in a Linit[®] (origin : Hanau,
15 Germany) type vessel containing 1.3 g of a standard powder detergent base
comprising 1% by weight of lipase (Lipolase[®] 100T ; origin : Novo Nordisk,
Denmark) and 260 ml water. The cotton swatch was washed for 30 min at
40°. It was then taken out of the vessel and rinsed with 3x200 ml of water. It
was then plunged for 5 min in 200 ml of water containing 0.6 g of a fabric
20 softener base which contained a certain percentage by weight, comprised
between 0.05 and 1%, of compound (I) according to the invention or, when
applicable, of the corresponding alcohol, aldehyde or ketone. The cotton
swatch was then spin-dried without having been rinsed, and dried on a
clothes line.

25 This method is equivalent to a washing in a real machine for washing 5 kg
of linen in approximately 20 l of water, with a rinsing of 4x20 l water, the
fabric softener being applied in the last rinsing water.

The fabric softener base used had the following composition :

30	<u>Ingredients</u>	<u>% by weight</u>
	Arquad 2 HT ¹⁾ (75%)	5.00
	Formalin (40%)	0.20
	Demineralized water	94.69
	Colouring agent ²⁾	0.11
35	Total	100.00

1) origin : Akzo, Holland

2) Colanyl Blau AR/10% sol. ; origin : Hoechst, Germany

In two separate tests, the cotton swatches were treated according to this general method, using as additive to the fabric softener respectively 9-decenyl hexadecanoate (1% by weight) in test A and 9-decenol (1% by weight) in test B.

The two swatches were submitted for a blind test evaluation 24 h after having been taken out of the Linitest[®] apparatus. The evaluation panel consisted of eight people, to whom had been given beforehand a smelling strip which had been dipped in 9-decen-1-ol, so as to render them familiar with the odour of this compound. The panel was then asked to smell the two cotton swatches above-mentioned and to indicate if they could identify the odour of 9-decen-1-ol in either of the swatches. Five amongst the eight members of the panel recognized the odour of this alcohol in the swatch treated in test A and indicated that this odour was very powerful, whereas only one of said members was able to identify the odour on the swatch treated in test B.

A further evaluation of the two swatches carried out by the same panel, under identical conditions, 24 h later, i.e. 48 h after taking the swatches out of the Linitest[®] apparatus, showed that none of the panel members could identify the odour of 9-decen-1-ol or the swatch of test B, whereas six of said members identified it without any hesitation on the swatch coming out of test A.

Similar results were obtained when the two compounds above-mentioned were present in the fabric softener at a rate of 0.5% by weight.

Example 5

30 Test on fabrics

Nine standard cotton swatches (32 g each), numbered from 1 to 9, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated in the fabric softener (at 1% by weight) and described in Table 1 hereinafter.

TABLE 1

COTTON SWATCH	SOFTENER ADDITIVE
1	9-decen-1-ol
2	9-decenyl octanoate
3	9-decenyl nonanoate
4	9-decenyl decanoate
5	9-decenyl undecanoate
6	9-decenyl dodecanoate
7	9-decenyl tridecanoate
8	9-decenyl tetradecanoate
9	9-decenyl pentadecanoate

24 H after taking the swatches out of the Linitest[®] machines, the nine cotton swatches were submitted for a blind evaluation to a panel consisting of ten individuals, whom, after having smelled 9-decen-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on any one of the swatches. The result of this evaluation is summarized on Table 2 hereafter (only categorical identifications were taken into account):

10

TABLE 2

COTTON SWATCH	Number of panel members having recognized the odour
1	0
2	10
3	8
4	3
5	6
6	10
7	8
8	7
9	9

In a similar test carried out 48 h after having taken the swatches out of the Linitest[®] machine, the members of the panel were still unanimous as regards swatch 1, i.e. they could not recognize the odour of 9-decen-1-ol on this swatch, whereas a majority judged it still very powerful on all the other swatches.

15

Example 6Test on fabrics

- 5 Six standard cotton swatches (32 g each), numbered from 1 to 6, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated in the fabric softener (at 1% by weight) and described in Table 3 hereinafter.

10 TABLE 3

COTTON SWATCH	SOFTENER ADDITIVE
1	9-decen-1-ol
2	bis(9-decenyl) oxalate
3	bis(9-decenyl) malonate
4	bis(9-decenyl) butanedioate
5	bis(9-decenyl) pentanedioate
6	bis(9-decenyl) hexanedioate

- 24 H after taking the swatches out of the Linitest[®] machines, the six cotton swatches were submitted for a blind evaluation to a panel consisting of ten
 15 individuals, whom, after having smelled 9-decen-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on any one of the swatches. The result of this evaluation is summarized on Table 4 hereafter (only categorical identifications were taken into account):

20 TABLE 4

COTTON SWATCH	Number of panel members having recognized the odour after 24 h
1	0
2	10
3	6
4	8
5	9
6	7

The same test, carried out 48 h after having taken the swatches out of the Linitest[®] machines, gave the results summarized in Table 5 hereinafter :

TABLE 5

COTTON SWATCH	Number of panel members having recognized the odour after 48 h
1	0
2	10
3	7
4	6
5	8
6	7

Example 710 Test on fabrics

Six standard cotton swatches (32 g each), numbered from 1 to 6, were separately treated, and in identical manner, as described in Example 4. The detergent's content in lipase was now 3% by weight and the amount of fabric softener added to the last rinsing water was now 1.2 g. The additive incorporated (at 1% by weight) into the fabric softener in each case is cited in the following Table 6 :

TABLE 6

COTTON SWATCH	SOFTENER ADDITIVE
1	(E)-3,7-dimethyl-2,6-octadien-1-ol
2	(E,E)-bis(3,7-dimethyl-2,6-octadienyl) oxalate
3	(E,E)-bis(3,7-dimethyl-2,6-octadienyl) malonate
4	(E,E)-bis(3,7-dimethyl-2,6-octadienyl) butanedioate
5	(E,E)-bis(3,7-dimethyl-2,6-octadienyl) pentanedioate
6	(E,E)-bis(3,7-dimethyl-2,6-octadienyl) hexanedioate

24 H after taking the swatches out of the Linitest[®] machines, the six cotton swatches were submitted for a blind evaluation to a panel consisting of ten individuals, whom, after having smelled (E)-3,7-dimethyl-2,6-octadien-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on any one of the swatches. The result of this evaluation is summarized on Table 7 hereafter (only categorical identifications were taken into account):

10 TABLE 7

COTTON SWATCH	Number of panel members having recognized the odour
1	2
2	7
3	7
4	9
5	7
6	8

Example 8

15

Test on fabrics

Two standard cotton swatches (32 g each), numbered 1 and 2, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated in the fabric softener and described in Table 8 hereinafter.

20

TABLE 8

COTTON SWATCH	SOFTENER ADDITIVE
1	3,7-dimethyl-6-octen-1-ol
2	3,7-dimethyl-6-octenyl hexadecanoate

25

24 H after taking the swatches out of the Linitest[®] machines, the two cotton swatches were submitted for a blind evaluation to a panel consisting of eleven individuals, whom, after having smelled (E)-3,7-dimethyl-6-octen-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on one or the other of the swatches. The result of this evaluation is summarized on Table 9 hereafter (only categorical identifications were taken into account):

TABLE 9

COTTON SWATCH	Number of panel members having recognized the odour
1	0
2	7

A similar test carried out with two cotton swatches treated with the same fabric softener base but containing only 0.5% by weight of the compounds reported in Table 8. The panel evaluation showed that none of the panel members was able to identify the odour of the alcohol on swatch 1, whereas 7 of the 11 members of the panel were certain that they could smell it on swatch 2.

Still analogous results were obtained with a dosage of 0.1% by weight of the above-mentioned compounds.

Example 9

Test on fabrics

Three standard cotton swatches (32 g each), numbered from 1 to 3, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated in the fabric softener (at 1% by weight) and described in Table 10 hereinafter.

TABLE 10

COTTON SWATCH	SOFTENER ADDITIVE
1	3-methyl-5-phenylpentan-1-ol
2	3-methyl-5-phenylpentyl octanoate
3	3-methyl-5-phenylpentyl hexadecanoate

- 24 H after taking the swatches out of the Linitest[®] machines, the three cotton swatches were submitted for a blind evaluation to a panel consisting of ten individuals, whom, after having smelled 3-methyl-5-phenylpentan-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on any one of the swatches. The result of this evaluation is summarized on Table 11 hereafter (only categorical identifications were taken into account):

TABLE 11

COTTON SWATCH	Number of panel members having recognized the odour
1	2
2	6
3	5

15

Example 10

Test on fabrics

- Three standard cotton swatches (32 g each) were treated separately following the general method described in Example 4, two of said swatches (1 et 2) using 9-decen-1-ol as additive to the softener, and swatch 3 using as additive 9-decenyl hexadecanoate, these additives having been added to the fabric softener in a concentration of 0.5% by weight.
- The three swatches were evaluated on a blind test by a panel of 12 individuals 24 h after having been taken out of the Linitest[®] machines. The members of the panel were asked if they could ascertain any difference amongst the swatches and, if applicable, whether they could qualify this

difference. The panel had previously smelled strips dipped in 9-decen-1-ol and was aware that an evaluation of the three cotton swatches with regard to the particular odour of this compound was expected.

As a result of this evaluation, 8 out of the 12 members of the panel chose swatch 3 as being the one olfactively distinct and indicated that it developed the characteristic odour of 9-decen-1-ol, whilst the other two swatches gave off no distinctive odour.

The same test was carried out with 1% of additive in the softener, instead of 0.5%. In this case, 9 of the 12 members of the panel chose once again swatch 3 as being the one developing the odour of the above-mentioned alcohol, and indicated that this odour was even more powerful than in the preceding test. On the other hand, the panel found that the other two cotton swatches still exhaled no distinctive odour.

Example 11

Test on fabrics

Two standard cotton swatches (32 g each), numbered 1 and 2, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated in the fabric softener, at a rate of 0.05% by weight, and described in Table 12 hereinafter.

TABLE 12

COTTON SWATCH	SOFTENER ADDITIVE
1	3,7-dimethyl-2,6-octadien-1-ol
2	3,7-dimethyl-2,6-octadienyl hexadecanoate

24 H after taking the swatches out of the Linitest[®] machines, the two cotton swatches were submitted for a blind evaluation to a panel consisting of twelve individuals, whom, after having smelled 3,7-dimethyl-2,6-octadien-1-ol on a smelling strip, had to indicate whether they recognized the odour of this alcohol on one or the other of the swatches. The result of this evaluation is summarized on Table 13 hereafter (only categorical identifications were taken into account):

TABLE 13

COTTON SWATCH	Number of panel members having recognized the odour
1	0
2	8

5

Example 12Test on fabrics

Two standard cotton swatches (32 g each), numbered 1 and 2, were
 10 separately treated, and in identical manner, as described in Example 4, but
 adding to the fabric softener 1% by weight of the additives cited in Table 14
 hereinafter.

TABLE 14

15

COTTON SWATCH	SOFTENER ADDITIVE
1	3,7-dimethyl-6-octenal
2	3,7-dimethyl-1,6-octadienyl octanoate

24 H after taking the swatches out of the Linitest[®] machines, the two
 cotton swatches were submitted for a blind evaluation to a panel consisting
 of eleven individuals, whom, after having smelled 3,7-dimethyl-6-octenal
 20 on a smelling strip, had to indicate whether they recognized the odour of
 this aldehyde on one or the other of the swatches. The result of this
 evaluation is summarized on Table 15 hereafter (only categorical
 identifications were taken into account):

25 TABLE 15

COTTON SWATCH	Number of panel members having recognized the odour
1	4
2	11

Example 13Test on fabrics

- 5 Two standard cotton swatches (32 g each), numbered 1 and 2, were separately treated, and in identical manner, as described in Example 4, the only changing element being the additive incorporated (at 1% by weight) in the fabric softener and described in Table 16 hereinafter.

10 TABLE 16

COTTON SWATCH	SOFTENER ADDITIVE
1	3-methyl-5-phenyl-1-pentanal
2	3-methyl-5-phenyl-1-pentenyl octanoate

- 24 H after taking the swatches out of the Linitest[®] machines, the two cotton swatches were submitted for a blind evaluation to a panel consisting of twelve individuals, whom, after having smelled 3-methyl-5-phenyl-1-pentanal on a smelling strip, had to indicate whether they recognized the odour of this aldehyde on one or the other of the swatches. The result of this evaluation is summarized on Table 17 hereafter (only categorical identifications were taken into account):

20

TABLE 17

COTTON SWATCH	Number of panel members having recognized the odour
1	2
2	8

25

Example 14Test on fabrics

- 30 Three standard cotton swatches (32 g each) were treated separately following the general method described in Example 4, two of said swatches

(1 et 2) using 2-methyl-1-undecanal as additive to the softener, and swatch 3 using as additive 2-methyl-1-undecenyl octanoate, these additives having been added to the fabric softener in a concentration of 0.1% by weight.

5 The three swatches were evaluated on a blind test by a panel of 12 individuals 24 h after having been taken out of the Linitest® machines. The members of the panel were asked if they could ascertain any difference amongst the swatches and, if applicable, whether they could qualify this difference. The panel had previously smelled strips dipped in 2-methyl-1-undecanal and was aware that an evaluation of the three cotton swatches
10 with regard to the particular odour of this compound was expected.

As a result of this evaluation, 10 out of the 12 members of the panel chose swatch 3 as being the one olfactively distinct and indicated that it developed the characteristic odour of 2-methyl-1-undecanal, whereas the other two swatches gave off no distinctive odour.

15

Example 15

Test on fabrics treated in a washing machine

20

Two standard batches of cotton textiles, each comprising 30 cotton swatches (32 g each), were put into two separate washing machines, each containing, in the detergent compartment, 130 g of a standard powder detergent base comprising 1% by weight of lipase (Lipolase® 100T; origin: Novo Nordisk,
25 Denmark). In the compartment intended for the fabric softener, there was put, in one of the cases, 100 g of a softener base such as described in Example 4, perfumed with 0.1% by weight of 2-methyl-1-undecanal, whereas the softener (100 g) put in the second machine contained 0.1% by weight of 2-methyl-1-undecenyl octanoate. The batches of swatches were then washed
30 with a normal washing cycle, at 40°, without prewash, and air dried.

The batches of swatches were evaluated on a blind test, 24 h after having been taken out of the machines, by a panel of 36 individuals. The latter had to indicate if they found any difference at all amongst the three batches of swatches, of which two were identical and had been treated with the
35 softener containing 2-methyl-1-undecanal (batches 1 and 2) and the third had been treated with 2-methyl-1-undecenyl octanoate (batch 3), without qualifying the nature of the distinctive character. In addition, they were

also asked to indicate whether any one of the batches possessed a more powerful odour.

Of the 36 panellists, 24 identified textile batch 3 as being the one distinct from the other two, from an olfactive point of view, and indicated that this
5 batch possessed a stronger odour than the other two.

An analogous test was carried out but with only two textile batches, batch A having been treated with a softener containing 0.05% by weight of 2-methyl-1-undecanal and batch B with a softener containing 0.05% by weight of the corresponding enol-ester, i.e. 2-methyl-1-undecenyl octanoate.

10 The panel of 36 individuals which evaluated batches A and B, without knowing the nature of the perfuming ingredients, had to indicate which of the two batches developed a stronger odour and which was preferred from the point of view of the perception of freshness and cleanliness of the linen.

15 According to the categoric opinion of 32 out of the 36 panellists, the textiles batch B was far more "perfumed" than batch A and was perceived as being cleaner, with a more fresh odour.

20

Example 16

Tests on fabrics treated in a washing machine

A base perfuming composition was prepared by admixing the following
25 ingredients :

	<u>Ingredients</u>	<u>% by weight</u>
	Benzyl acetate	30
	1,1-Dimethyl-2-phenylethyl acetate	25
30	10-Undecenal	3
	Mexylcinnamic aldehyde	150
	Methyl anthranilate dist.	10
	Cedroxyde ® 1)	40
	10%* Ambrox ® 2) DL	2
35	Verdyl acetate 3)	30
	10% * α-Damascone	3
	Dihydromyrcenol 4)	80
	10%* Dynascone ® 5)	2



	Eugenol	7
	Exaltex ® 6)	50
	Galbex ® 7) 183	8
	Geranyl nitrile	1
5	10% ** Indol	5
	Italia ® 8)	20
	Isoeugenol	3
	Lilial ® 9)	50
	Linalol	30
10	Lorysia ® 10)	50
	Mayol ® 11)	10
	Hedione ® 12)	50
	10% * Rose oxide 13)	5
	Phenethylol	60
15	Polysantol ® 14)	3
	Orange essential oil	40
	Benzyl salicylate	30
	Scentenal ® 15)	3
	Terpineol	25
20	Veloutone 16)	2
	Verdox ® 17)	20
	β-Naphtol methyl-ether	2
	Zestover ® 18)	1
25	Total	<hr/> 850

* in dipropyleneglycol (DIPG)

** in triethanolamine

- 1) trimethyl-13-oxabicyclo[10.1.0]trideca-4,8-diene ; origin : Firmenich SA, Geneva, Switzerland
- 2) dodecahydro-3,6,6,9a-tetramethyl-naphto[2,1-b]furan ; origin : Firmenich SA, Geneva, Switzerland
- 3) tricyclo[5.2.1.0^{2,6}]dec-3-en-8-yl acetate ; origin : Givaudan-Roure, Vernier, Switzerland
- 35 4) 2,6-dimethyl-7-octen-2-ol ; origin : International Flavors & Fragrances, USA
- 5) 1-(5,5-dimethyl-1-cyclohex-1-yl)-4-penten-1-one ; origin : Firmenich SA, Geneva, Switzerland

- 6) oxacyclohexadecan-2-one ; origin : Firmenich SA, Geneva, Switzerland
- 7) origine : Firmenich SA, Geneva, Switzerland
- 8) methylionone ; origin : Firmenich SA, Geneva, Switzerland
- 9) 3-(4-tert-butyl-1-phenyl)-2-methylpropanal ; origin : Givaudan-Roure,
5 Vernier, Switzerland
- 10) 4-(1,1-dimethylethyl)-cyclohexanol acetate ; origin : Firmenich SA,
Geneva, Switzerland
- 11) cis-4-(1-methylethyl)-cyclohexanemethanol ; origin : Firmenich SA,
Geneva, Switzerland
- 10 12) methyl ester of 3-oxo-1-pentyl-cyclopentaneacetic acid ; origin :
Firmenich SA, Geneva, Switzerland
- 13) origin : Firmenich SA, Geneva, Switzerland
- 14) 3,3-dimethyl-5-(2,2,3-trimethyl-3-cyclopenten-1-yl)-4-penten-2-ol ;
origin : Firmenich SA, Geneva, Switzerland
- 15 15) octahydro-5-methoxy-4,7-methano-1H-indene-2-carboxaldehyde ;
origin : Firmenich SA, Geneva, Switzerland
- 16) 2,2,5-trimethyl-5-pentyl-cyclopentanone ; origin : Firmenich SA,
Geneva, Switzerland
- 17) 2-tert-butyl-cyclohexanyl acetate ; origin : International Flavors &
20 Fragrances, USA
- 18) 2,4-dimethyl-3-cyclohexene-1-carbaldehyde ; origin : Firmenich SA,
Geneva, Switzerland

Two perfuming compositions were prepared with this base composition, by
25 adding thereto 150 parts by weight of geraniol or 3,7-dimethyl-2,6-
octadienol (composition A), respectively 150 parts by weight of 3,7-
dimethyl-2,6-octadienyl hexadecanoate (composition B).

These two perfuming compositions were then added, at the rate of 0.6% by
weight, to a non-perfumed fabric softener base to prepare two samples, A
30 and respectively B, of a perfumed softener.

In two identical washing machines, there were treated two standard batches
of textiles, each formed of 1400 g of varied linen, comprising cotton
swatches, cotton textiles and textiles in a cotton/polyester mixture. Each
machine was charged with 130 g of a standard unperfumed detergent base
35 containing 1% of lipase (Lipolase ® 100T). One of the machines was also
charged with 100 g of sample A of the softener, comprising geraniol,
whereas to the second machine there were added 100 g of softener sample
B.

The linen was then washed at 40° in a normal washing cycle, without prewash, and dried to air for 24 h.

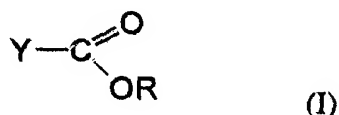
5 Three standard batches of textiles, of which two were identical and had been treated with sample A and the third with sample B, were then evaluated on a blind test by a panel of 20 individuals, who had to identify the batch of textiles possessing a distinct odour from that of the other two, without qualifying the nature of the distinctive character. The panellists also had to indicate which of the textile batches was more perfumed.

10 As a result of this test, 14 panellists perceived a distinction in the odour of the textile batch treated with the fabric softener sample B, while 10 out of these 14 indicated that this same batch was the more perfumed.

When the evaluation was repeated after 48 h of drying to air, analogous results were obtained.

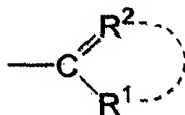
The claims defining the invention are as follows:-

1. A process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, said process being characterized in that said detergent and optionally said fabric softener contains a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener is obligatorily used and contains a compound of formula

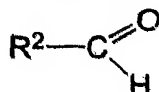


wherein

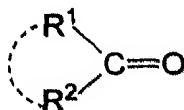
- 10 a. R represents a radical derived from a fragrant alcohol of formula ROH and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6; or
- 15 b. Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula



wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from a fragrant aldehyde of formula



- 20 or R² represents an alkylidene radical and R¹ an alkyl radical, R¹ and R² being then derived from a fragrant ketone of formula



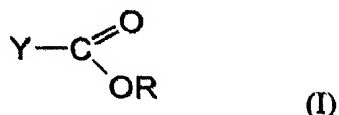
and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.

- 25 2. A process for perfuming fabrics washed in the presence of a lipase-containing detergent, which process comprises the treatment of said fabrics, after the washing cycle, with a fabric softener containing a compound of formula (I) as defined in claim 1.

3. A process for washing fabrics, comprising a washing cycle in the presence of a lipase-containing detergent, optionally followed by a treatment with a fabric softener, said process being characterized in that said detergent and optionally said fabric softener



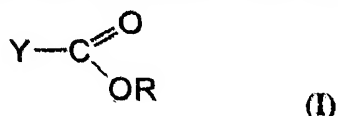
contains a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener is obligatorily used and contains a compound of formula



5 as defined in claim 1.

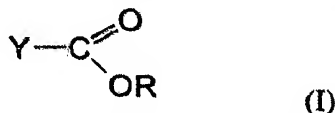
4. A process for washing fabrics, comprising a washing cycle in the presence of a lipase-containing detergent, followed by the treatment of said fabrics with a fabric softener containing a compound of formula (I) as defined in claim 1.

5. A process for prolonging the diffusion effect of the odour developed by fabrics
10 and which is characteristic of a fragrant alcohol of formula ROH, said process being characterized in that the fabrics undergo a washing cycle in the presence of a lipase-containing detergent and, optionally, a subsequent treatment with a fabric softener, said detergent and optionally said fabric softener containing a compound of formula (I),
15 said fabric softener being obligatorily used and containing a compound of formula



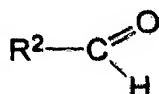
wherein R represents a monovalent radical derived from said fragrant alcohol, and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6.

20 6. A process for prolonging the diffusion effect of the odour characteristic of a fragrant alcohol of formula ROH and which is developed by fabrics washed with a lipase-containing detergent, said process being characterized in that said fabrics, after the washing cycle, are treated with a fabric softener containing a compound of formula



25 wherein R represents a monovalent radical derived from said fragrant alcohol, and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6.

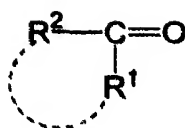
7. A process for prolonging the diffusion effect of the odour developed by fabrics and which is characteristic of a fragrant aldehyde of formula



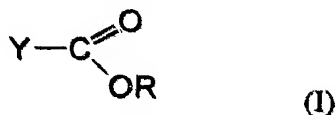
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or of a fragrant ketone of formula

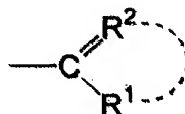




said process being characterized in that the fabrics undergo a washing cycle in the presence of a lipase-containing detergent and, optionally, a subsequent treatment with a fabric softener, said detergent and optionally said fabric softener containing a compound of formula (I) as specified below, or, in case said detergent does not contain said compound of formula (I), said fabric softener being obligatorily used and containing a compound of formula



wherein Y represents a C₇ to C₂₄, linear or branched, saturated or unsaturated alkyl radical, and R represents a group of formula

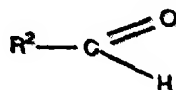


wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from said fragrant aldehyde, or R² represents an alkylidene radical and R¹ an alkyl radical, R¹ and R² being then derived from said fragrant ketone and, optionally, being part of a ring such as indicated by the

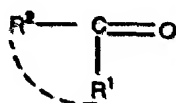


dotted line which contains from 5 to 18 carbon atoms and can be substituted.

8. A process for prolonging the diffusion effect of the odour characteristic of a fragrant aldehyde of formula



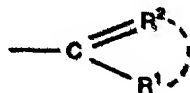
or of a fragrant ketone of formula



developed by fabrics washed with a lipase-containing detergent, said process being characterized in that said fabrics, after the washing cycle, are treated with a fabric softener containing a compound of formula



wherein Y represents a C₇ to C₂₄, linear or branched, saturated or unsaturated alkyl radical, and R represents a group of formula



wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from said fragrant aldehyde, or R² represents an alkylidene radical and R¹ an alkyl radical, R¹ and R² being then derived from said fragrant ketone and, optionally, being part of a ring such as indicated by the dotted line which contains from 5 to 18 carbon atoms and can be substituted.

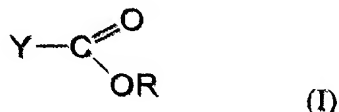
9. A process according to one of claims 1 to 6, comprising the addition of a compound of formula (I) wherein R is the 9-decen-1-yl, 3,7-dimethyl-2,6-octadien-1-yl, 3,7-dimethyl-6-octen-1-yl or 3-methyl-5-phenylpentyl radical.

10. A process according to any one of claims 1 to 4, 7 or 8, comprising the addition of a compound of formula (I) wherein R is the 3,7-dimethyl-1,6-octadienyl, 3-methyl-5-phenyl-1-pentenyl or 2-methyl-1-undecenyl radical.

11. A process according to any one of claims 1 to 10, comprising the addition of a compound of formula (I) wherein Y is a C_{12} to C_{24} linear alkyl radical.

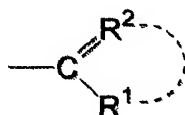
12. A laundry composition comprising a detergent or a fabric softener base and a compound of formula (I) as defined in claim 1.

13. A compound of formula

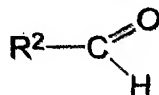


10 wherein

- a. R represents a radical derived from a fragrant alcohol of formula ROH and Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical, or a $-(\text{CH}_2)_n\text{COOR}$ group wherein R is defined as above and n is an integer from 0 to 6; or
- 15 b. Y represents a C_7 to C_{24} linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula

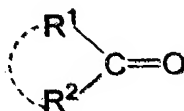


wherein, either R^1 represents hydrogen and R^2 represents an alkylidene radical derived from a fragrant aldehyde of formula



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or R^2 represents an alkylidene radical and R^1 an alkyl radical, R^1 and R^2 being then derived from a fragrant ketone of formula



and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.

14. A compound according to claim 13, wherein R is the 9-decen-1-yl, 3,7-dimethyl-2,6-octadien-1-yl, 3,7-dimethyl-6-octen-1-yl, 3-methyl-5-phenylpentyl, 3,7-dimethyl-1,6-octadienyl, 3-methyl-5-phenyl-1-pentenyl or 2-methyl-1-undecenyl radical.

15. A compound according to claim 14 wherein R is the 9-decen-1-yl or 3,7-dimethyl-2,6-octadien-1-yl radical.



16. A compound according to claim 13, claim 14 or claim 15 wherein Y is a C₁₂ to C₂₄ linear alkyl radical.

17. A perfuming composition containing a compound of the formula (I) as defined in claim 1 together with a current perfuming ingredient, solvent or adjuvant.

5 18. A process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, substantially as hereinbefore described with reference to any one of the Examples.

19. An ester derived from a fragrant alcohol, substantially as hereinbefore described with reference to any one of the Examples.

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Dated 22 September 1997
Firmenich SA

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Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON



17. A process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, substantially as hereinbefore described with reference to any one of the Examples.

18. A process for prolonging the diffusion effect of the odour developed by fabrics and which is characteristic of a fragrant alcohol, substantially as hereinbefore described with reference to any one of the Examples.

19. A process for prolonging the diffusion effect of the odour characteristic of a fragrant alcohol, substantially as hereinbefore described with reference to any one of the Examples.

20. A process for prolonging the diffusion effect of the odour developed by fabrics and which is characteristic of a fragrant aldehyde, substantially as hereinbefore described with reference to any one of the Examples.

21. An ester derived from a fragrant alcohol, substantially as hereinbefore described with reference to any one of the Examples.

Dated 6 April, 1995
Firmenich SA

Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON



ABSTRACT

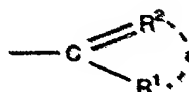
We describe a process for perfuming fabrics washed in the presence of a lipase-containing detergent and, optionally, subsequently treated with a fabric softener, said process being characterized in that said detergent and/or said fabric softener contains a compound of formula



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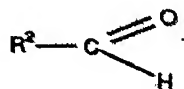
wherein

- a. R represents a radical derived from an fragrant alcohol of formula ROH and Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical, or a -(CH₂)_nCOOR group wherein R is defined as above and n is an integer from 0 to 6 ; or
- b. Y represents a C₇ to C₂₄ linear or branched, saturated or unsaturated alkyl radical and R represents a group of formula



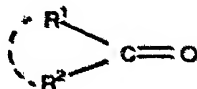
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wherein, either R¹ represents hydrogen and R² represents an alkylidene radical derived from a fragrant aldehyde of formula



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or R² represents an alkylidene radical and R¹ an alkyl radical, R¹ and R² being then derived from an fragrant ketone of formula



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and, optionally, being part of a ring such as indicated by the dotted line which contains 5 to 18 carbon atoms and can be substituted.



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The process has the advantage of providing an effect of slow diffusion ("slow release") of said fragrant alcohol, aldehyde or ketone, thus prolonging the odour effect of the latter on fabrics.



INTERNATIONAL SEARCH REPORT

International Application No.
PCT/IB 94/00228

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D3/50 C11D3/386 C07C69/007

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C11D C07C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 430 315 (UNILEVER NV ; UNILEVER PLC (GB)) 5 June 1991 cited in the application see the whole document -----	1-16
A	EP,A,0 397 245 (PROCTER & GAMBLE) 14 November 1990 cited in the application -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *Z* document member of the same patent family

Date of the actual completion of the international search

9 December 1994

Date of mailing of the international search report

15 12 94

Name and mailing address of the ISA

European Patent Office, P.B. 3818 Patentlaan 2
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Authorized officer

Delzant, J-F

INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/IB 94/00228

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0430315	05-06-91	AU-B- 648039	14-04-94
		AU-A- 6319890	11-04-91
		JP-A- 3131696	05-06-91

EP-A-0397245	14-11-90	AU-A- 5491290	29-11-90
		CA-A- 2015736	11-11-90
		CN-A- 1047336	28-11-90
		JP-A- 3033196	13-02-91

RAPPORT DE RECHERCHE INTERNATIONALE

Dem. Internationale No
PCT/IB 94/00228

A. CLASSEMENT DE L'OBJET DE LA DEMANDE
CIB 6 C11D3/50 C11D3/386 C07C69/007

Selon la classification internationale des brevets (CIB) ou à la fois selon la classification nationale et la CIB

B. DOMAINES SUR LESQUELS LA RECHERCHE A PORTE

Documentation minimale consultée (système de classification suivi des symboles de classement)
CIB 6 C11D C07C

Documentation consultée autre que la documentation minimale dans la mesure où ces documents relèvent des domaines sur lesquels a porté la recherche

Base de données électronique consultée au cours de la recherche internationale (nom de la base de données, et si cela est réalisable, termes de recherche utilisés)

C. DOCUMENTS CONSIDERES COMME PERTINENTS

Catégorie	Identification des documents cités, avec, le cas échéant, l'indication des passages pertinents	no. des revendications visées
A	EP,A,0 430 315 (UNILEVER NV ; UNILEVER PLC (GB)) 5 Juin 1991 cité dans la demande voir le document en entier ---	1-16
A	EP,A,0 397 245 (PROCTER & GAMBLE) 14 Novembre 1990 cité dans la demande -----	

☐ Voir la suite du cadre C pour la fin de la liste des documents

☒ Les documents de familles de brevets sont indiqués en annexe

*** Catégories spéciales de documents cités:**

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- *T* document ultérieur publié après la date de dépôt international ou la date de priorité et n'appartenant pas à l'état de la technique pertinent, mais cité pour comprendre le principe ou la théorie constituant la base de l'invention
- *X* document particulièrement pertinent; l'invention revendiquée ne peut être considérée comme nouvelle ou comme impliquant une activité inventive par rapport au document considéré isolément
- *Y* document particulièrement pertinent; l'invention revendiquée ne peut être considérée comme impliquant une activité inventive lorsque le document est associé à un ou plusieurs autres documents de même nature, cette combinaison étant évidente pour une personne du métier
- *Z* document qui fait partie de la même famille de brevets

Date à laquelle la recherche internationale a été effectivement achevée

9 Décembre 1994

Date d'expédition du présent rapport de recherche internationale

15.12.94

Nom et adresse postale de l'administration chargée de la recherche internationale
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Fax (+31-70) 340-3016

Fonctionnaire autorisé

Delzant, J-F

RAPPORT DE RECHERCHE INTERNATIONALE

Renseignements relatifs aux membres de familles de brevets

Dem Internationale No

PCT/IB 94/00228

Document brevet cité au rapport de recherche	Date de publication	Membre(s) de la famille de brevet(s)	Date de publication
EP-A-0430315	05-06-91	AU-B- 648039	14-04-94
		AU-A- 6319890	11-04-91
		JP-A- 3131696	05-06-91

EP-A-0397245	14-11-90	AU-A- 5491290	29-11-90
		CA-A- 2015736	11-11-90
		CN-A- 1047336	28-11-90
		JP-A- 3033196	13-02-91
